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Lidia GURAU - Mihaela CAMPEAN - Mihai ISPAS

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KEYNOTE ADDRESSES

FORMALDEHYDE IN WOOD: WHERE DOES IT COME FROM?

Mark IRLE, Thérèse FRIEDRICH, Marie LABISTE and Tristan RICHARD
LIMBHA, Ecole Supérieure du Bois, rue Christian Pauc, 44306 Nantes, France
Tel: +33 240 18 12 12 Email: mark.irle@ecoledubois.fr

Abstract

The research described here attempts to better understand the origin of the formaldehyde emitted by Pinus radiata. Previous research suggests that wood generates formaldehyde as a by-product from some degrading chemistry. It is suggested that at least some of the formaldehyde that is released from wood has been previously adsorbed by the wood. This paper compares the formaldehyde emission from natural and acetylated Pinus radiata; the latter having a lower equilibrium moisture content. The results indicate that acetylated wood emits less formaldehyde than untreated wood in tests where the specimens approach the oven-dry condition by the end of the test. It is also observed that the quantity of formaldehyde emitted when a test is repeated on a specimen is lower than the initial emission. It is proposed that these observations can be explained if it is accepted that part of the formaldehyde emission from wood is derived by the prior adsorption of formaldehyde by wood. It is estimated that at least 50% of the formaldehyde observed is due to prior adsorption.

INTRODUCTION

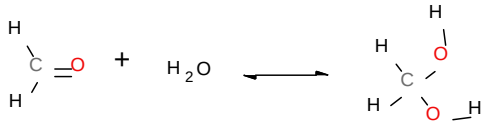
In June of 2004, the International Agency for Research into Cancer (IARC), which is part of WHO (World Health Organization), raised the cancer rating of formaldehyde from category 2A (probably carcinogenic to humans) to Category 1B (carcinogenic to humans based on animal data). This decision was confirmed in 2009. As a consequence, the European REACH Committee reclassified formaldehyde as a category 1B carcinogen in December 2013.

Various researchers have observed that natural wood releases formaldehyde see, for example, Meyer and Boehme (1997), Schäfer and Roffael (2000) and Weigl et al., (2009). In all cases, the levels observed were low, but, detectable. The small amounts of formaldehyde emitted do not pose any health risk. Even so, the fact that wood is seen to emit formaldehyde may have an impact in its use for interior applications because of the increasingly stringent indoor air quality regulations across the World.

Marutzky and Roffael (1977) proposed various mechanisms by which formaldehyde might be generated by the polymers and extractives present in wood, see Figure 1. It is argued, however, that formaldehyde, which has been previously adsorbed by the wood, makes up a part of the formaldehyde observed in tests. This seems possible because wood is hygroscopic; it will change its moisture content as a function of its surrounding temperature, humidity and pressure. Wood is also able to adsorb and desorb other polar molecules like formaldehyde. In a very simple experiment Irle et al. (2008) demonstrated that wood will readily adsorb formaldehyde from the atmosphere and then desorb it during a formaldehyde emission test.

Formaldehyde is omnipresent. Previous research indicates that the concentration of formaldehyde in buildings is somewhat higher than that outside. Indoor values range from <30 µg/m^3 to over 300 µg/m^3 with a mean of 58.6 µg/m^3, whereas outside the concentration is typically less than 10 µg/m^3 (Kraus *et al.* 1991). Wood stored indoors is likely to adsorb and desorb both water and formaldehyde. Could it be possible that formaldehyde accumulates in wood? Certainly, formaldehyde is a very reactive molecule and polar.

When formaldehyde is in water it forms methylene glycol (methanediol):

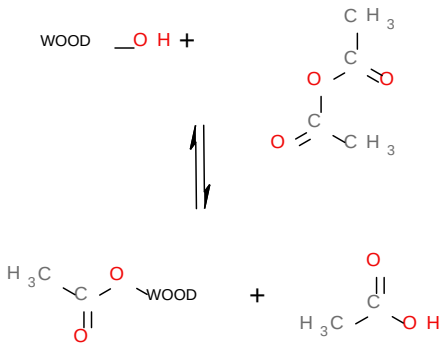


It is suggested that a proportion of formaldehyde in wood is actually in the form of methylene glycol. Some formaldehyde, on the other hand, will form hydrogen bonds with the plethora of hydroxyl groups present in wood and some may even react with the hydroxyls to form hemi-acetals.
Logically, three scenarios could explain the emission of formaldehyde by wood:

1. Wood generates the majority of formaldehyde as a by-product from some degrading chemistry
2. The majority of the formaldehyde that is released from wood has been previously adsorbed by the wood
3. Both mechanisms play a role

Current thinking suggests that scenario 1 is the main mechanism. If true, then this could eventually lead to regulations that limit the quantity of wood in the indoor environment in terms of *x* m^2/m^3 of room. If, on the other hand, most of the formaldehyde comes from an adsorption/desorption mechanism, then wood can be used with impunity. The research described here attempts to better understand the origin of the formaldehyde emitted by wood.

The approach described in this paper is to compare the formaldehyde emission of untreated and acetylated *Pinus radiata*. The acetylated wood will have a lower equilibrium moisture content, i.e. it is less hygroscopic, and so if scenario 2 has any effect, then one would expect the formaldehyde emission from acetylated wood to be lower than the untreated. Acetylation reduces the hygroscopicity of wood via chemical modification of the hydroxyl groups in wood (Rowell 2013):



acetic acid

It is proposed that after acetylation wood will be less able to adsorb formaldehyde and, therefore, emit less during a formaldehyde emission test. Of course, the acetylation process may also change the ability of wood to generate formaldehyde. This paper describes an experiment that compares the emission of formaldehyde from natural and acetylated *Pinus radiata*.

MATERIALS & METHODS

Pinus radiata was supplied by Accsys Group from their manufacturing plant in Arnhem, Holland. All pieces were selected from the same batch in order to minimise any differences between acetylated and untreated wood. All wood was stored in a laboratory without control of atmospheric conditions.

The pieces were numbered by Accsys so that treated and untreated pieces could be matched. The original dimensions were 25 x 190 x 800 mm (thickness x width x length). Two of pieces of acetylated wood were selected together with their untreated pair. These were crosscut in half and then sawn into 40 mm wide pieces thus giving specimens with nominal dimensions of 25 x 40 x 395 mm.

Formaldehyde emission was measured using EN ISO 12460-3 "Wood-based panels — Determination of formaldehyde release — Part 3: Gas analysis method". The test procedure involves heating the specimens with dry air at 60 °C and so, of course, the specimens lose weight during the test. Unlike the standard, the results are expressed in µg formaldehyde/100 g of specimen.

After a number of initial tests, it was decided to further reduce the thickness of the specimens by cutting them in half, length ways through the thickness, to give specimens approximately 9 x 40 x 395 mm. In addition, it was decided to repeat formaldehyde emission tests on some specimens. Consequently, these specimens were crosscut in half to reduce their length so that they could be rehumidified in desiccators. Each pair of halves were tested together to approximate the previous specimen size. Rehumidifcation was achieved by placing 200 mL of deionised water in an evaporation dish in the desiccator. The specimens were reconditioned for approximately 48 hours before retesting. The formaldehyde content of the deionised water was also measured after the conditioning period.

RESULTS

The results in Table 1 present the formaldehyde emission values in terms of µg/100 g of conditioned wood (not oven dry). No statistically significant difference was observed between emission rates of untreated and acetylated for the 24 mm thick specimens. This is probably because specimens

do not dry out completely during the test. The average moisture contents of acetylated wood was 3.0%, whereas untreated had an average of 10.4%. At the end of the tests their respective moisture contents were 1.8% and 7.8%. Reducing the thickness to a nominal 9 mm revealed a significant difference, at a 99% confidence, in emission rates between untreated and acetylated wood. Tasooji et al (2017), using a completely different test method, found similar emission levels for heated *P. radiata*.

Table 1

The formaldehyde emissions observed from P. radiata

Thickness (mm)	UNTREATED			ACETYLATED		
	No.	Mean (µg/100g)	Standard deviation	No.	Mean (µg/100g)	Standard deviation
24	3	33	5.6	3	33	7.6
9	11	54	11.4	10	41	7.8

These results are probably linked to the moisture content changes during the test. The thinner have a greater surface area to volume and so they increase in temperature more rapidly and dry out more quickly. The average end moisture contents at the end of the tests were 5.1% for untreated and 0.7% for acetylated specimens.

It is well known that formaldehyde emissions are positively correlated to moisture content. For example, formaldehyde test methods like EN ISO 12460-5 contain a correction factor to normalise the observed formaldehyde concentrations to a moisture content of 6.5% and that specimens should have moisture content of between 3 and 10%

It is interesting to note that none of the chemical reactions proposed by Marutzky and Roffael (1977) nor Schäfer and Roffael (2000) include water as a reactant, rather they propose that water could be produced as part of the polymer breakdown reactions, see Figure 1. One mechanism proposed involves the dehydration of hexoses to form oxymethylfurfurals which then degrade to furfural and formaldehyde. It would seem that the generation of formaldehyde by hemicelluloses would be favoured by drying conditions and so one would expect more formaldehyde to be generated at higher than ambient temperatures. In fact, the reverse is seen in the EN ISO 12460-5 where emission rate falls as the test progresses, i.e. as the specimens dry. Likewise none of the lignin degradation routes proposed require water as a reactant, an example is shown in Figure 1. Therefore, purely from a chemical reaction point of view, the presence of water or not should not have an impact on the production of formaldehyde by wood. Therefore, one could conclude that the breakdown reactions should be independent of moisture content. The fact that formaldehyde emission falls, implies that the emission of formaldehyde by a chemical reaction is less important.

Given that Irle et al. (2008) demonstrated that wood can adsorb formaldehyde then the total formaldehyde emission from a wood object should consist of that which has be previously adsorbed and then desorbed during the test (E_{des}) and that which might be generated by breakdown of wood polymers (E_{chem}), see Eq. 1. What is not known at this stage is the relative proportions of E_{des} and E_{chem}.

$$E_{total} = E_{des} + E_{chem} \qquad \text{(Eq. 1)}$$

Clearly the chemical properties of acetylated wood are different to untreated wood and so one would expect E_{chem} of acetylated wood to be different to untreated. From data presented by Rowell (2014) it is known that little of the cellulose present will have reacted with the acetic anhydride. It is estimated that about 80% of OH groups in the lignin and 30% of OH in hemicelluloses would have been modified by acetic anhydride at the weight percent gain (WPG) used in commercial products. This could have an impact on wood's ability to generate formaldehyde and this may also explain the differences in emission levels observed between untreated and acetylated wood.

The EMC values of acetylated and untreated are quite different and so E_{des} are also expected to be different. The values of E_{des} and E_{chem} for both cases cannot be resolved with the information thus far collected.

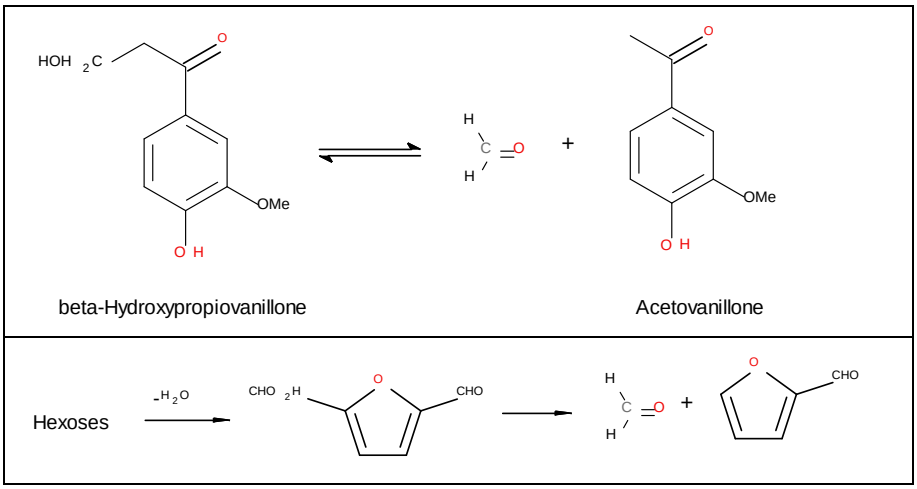


Fig. 1.
Two examples of the possible reaction mechanisms that could generate formaldehyde in wood as proposed by Marutzky and Roffael (1977) and Schäfer and Roffael (2000).

Table 2 shows the formaldehyde relative to the mass loss observed during the test. It would seem that the amount of formaldehyde emitted per gram of mass loss is higher for the acetylated wood. This is somewhat unexpected as one might expect the concentration of formaldehyde to be the same due to vapour pressure laws. The mass loss is largely due to moisture loss, but, other volatiles associated with the extractives present and certainly acetic acid in the acetylated wood also contribute to the loss.

Table 2
The formaldehyde emission relative to the mass loss of the specimens during the tests

Thickness (mm)	UNTREATED			ACETYLATED		
	No.	Mean (µg/g loss)	Standard deviation	No.	Mean (µg/g loss)	Standard deviation
24	3	11	1.4	3	23	2.5
9	11	11	2.2	10	17	3.2

An alternative approach to determine the relative importance of E_{des} and E_{des} was tried. This involved repeat testing of particular samples. Once a specimen had completed a test it was sealed in a desiccator with deionised water as described in the methods. The idea is that the specimens would rehumidify but not have the opportunity to adsorb formaldehyde from the atmosphere. Any differences in subsequent emission tests should be more closely linked to changes in E_{des} rather than E_{chem}. The data in Table 3 show the emission levels fall with each repetition of the test. The 9R0 specimens are equivalent to the 9 mm thick specimens shown in Table 1. The higher emission rates of these specimens is thought to be due to the fact that the specimens had been crosscut in half thus doubling to amount of transverse face, which is likely to aid drying and escape of volatiles like formaldehyde. To date only two repetitions have been conducted to date and so the difference may be due to chance. Each repetition of the test brings about reduced emission levels. For untreated wood the trend is linear and for the acetylated asymptotic.

Table 3
Formaldehyde emission levels observed after repeat measurements on the same samples

Thickness (mm)	UNTREATED			ACETYLATED		
	No.	Mean (µg/100g)	Standard deviation	No.	Mean (µg/100g)	Standard deviation
9R0	2	68	10.3	2	50	9.2
9R1	2	64	7.5	2	36	4.1
9R2	2	50	7.2	2	24	10.5
9R3	2	46	5.9	1	28	

Table 4 shows how the formaldehyde emission relative to the observed mass loss, changed with each repetition of test. It is clear that for both the acetylated and untreated wood that the

formaldehyde emission decreases with each repetition of the test. The reconditioning step caused slightly higher moisture contents at the start of the repeat tests; this should increase the formaldehyde emission and so the reductions observed are even more remarkable. It may be coincidence, but, it is seems that µg/g loss stabilises at around 7 µg/g loss. The chemistry of the specimens is not thought to change a great deal between repetitions of the cycle. If so, then it the 7 µg/g loss might be due to chemical reactions (E_{chem}) like that proposed by Marutsky and Roffael (1977), and therefore the values from the initial tests would indicate that E_{des} would be equal to about 6 µg/g loss for the untreated wood and 13 µg/g loss for the acetylated, i.e. E_{des} equates to between a little over 50% to 60% for the untreated and acetylated respectively.

Table 4

The quantity of formaldehyde emitted relative to the mass loss observed after repeat measurements on the same samples

Thickness (mm)	Final HCHO conc. in water	UNTREATED					ACETYLATED				
		No.	MC start	MC end	Mean (µg/g loss)	Standard deviation	No.	MC start	MC end	Mean (µg/g loss)	Standard deviation
9R0		2	10.1%	4.9%	13	1.9	2	3.0%	0.4%	20	3.6
9R1	59	2	12.8%	4.4%	10	1.3	2	4.0%	0.5%	11	1.5
9R2	52	2	13.3%	4.5%	7	1.5	2	4.2%	0.5%	7	3.2
9R3	45	2	12.7%	4.3%	7	0.9	2	4.2%	0.5%	8	

The quantity of formaldehyde found in the deionised water at the end of each conditioning period also fell with each repeat test, see first column of Table 4. Given the mass of wood present, it is suggested that this fall is not due to significant changes in the chemistry of the wood. Rather the reduction derives from a sort of "washing" out of free formaldehyde from the wood. It is thought that formaldehyde does not readily diffuse out of wood once it is dry. Therefore, formaldehyde remaining at the end of an emission test is likely to remain in the wood and, hence, the low emissions observed in the final hours of the test. As the moisture content of the specimens increases the formaldehyde is more able to diffuse. Following concentration laws, some of the formaldehyde will diffuse out of the wood and into the deionised water. If wood does not generate formaldehyde so that all the formaldehyde observed has been previously adsorbed, then given enough cycles, the formaldehyde emission rate should fall to zero. What is needed, therefore, are more repetitions in two senses: one terms of the number of specimens tested and the other in terms of number of cycles a specimen is tested.

CONCLUSIONS

The results clearly show that acetylated wood emits less formaldehyde than untreated wood in tests where the specimens approach the oven-dry condition by the end of the test. It is also observed that the quantity of formaldehyde emitted when a test is repeated on a specimen is lower than the initial emission. It is proposed that these observations can be explained if it is accepted that part of the formaldehyde emission from wood is derived by the prior adsorption of formaldehyde by wood. It is estimated that at least 50% of the formaldehyde observed is due to prior adsorption. Additional tests are required to confirm this observation. Ongoing work will increase the number of repetitive tests and also observe formaldehyde emissions after specimens have been reconditioned in the laboratory for some months.

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MOISTURE DYNAMICS DEFINING SERVICE LIFE PERFORMANCE OF WOOD PRODUCTS

Joris VAN ACKER
Ghent University (UGent), Laboratory of Wood Technology (Woodlab)
Coupure links 653, 9000 Ghent, Belgium
Tel: 0032 9 2646120, Fax: 0032 9 2646233, E-mail: Joris.VanAcker@UGent.be

Abstract

Material resistance of wood products against fungal decay has long been considered as a direct derivative of the biological durability, merely the biocidal protection against organisms able to degrade wood. When targeting those applications where moisture content can be controlled by the mere composition or structure of the wood products the service life of a commodity can be significantly impacted. This allows to expect a considerable service life even for so-called non durable wood material.The service life of wood products in exterior applications and in particular in ground contact has since long been defined by a durability class. This durability class is defined mainly by the impact of decaying organisms on the mechanical integrity over time. In ground contact the wood moisture content is nearly always sufficient to sustain both Basidiomycetes induced decay (white rot and/or brown rot) as well as soft rot. However, when exposing wood products outdoors without direct interaction with water or soil, the so-called use class 3 according to EN 335, moisture dynamics become relevant in addition to the inherent biological durability based on biocidal components. Time of wetness can be regarded as a tool to illustrate the possibility to degrade or not by fungi. This is valid for the properties of a wood species, but also for the different wood protecting methods to enhance the service life. Not only the introduction of biocidal actives as done by means of wood preservation technology, but also different wood modifying techniques as well hydrophobation and the application of coatings can be considered valid to prolong service life of wood products.

The role of inherent wood durability of wood species and their origin as well as options to enhance both durability as moisture dynamics allow the wood industry to provide a range of product types and performance levels. This variability in properties from biological origin in combination with man-made transformations allows the end user to select from a range of alternatives to accomplish fit for purpose criteria defined by limit states. These have been identified as simple classification systems in the past but are now evolving toward model based approaches combining resistance to biological degradation and moisture dynamics. European standardization within CEN TC 38 on wood durability is working on this topic and test methodology as well as decision support mechanisms are under discussion. Meanwhile also the forum on how to enhance wood properties is working on this renewed concept, e.g. the International Research Group on Wood Protection (IRGWP) and the European Conference on Wood Modification (ECWM). Although the approach to match performance of wood products with material characteristics seems straightforward several additional parameters seem to interact and require additional stochastic modelling related to different commodities, overall and local climate, design, biological hazard, presence and probability of decay organisms, time of wetness, etc. Extra tools are being developed to understand such parameters. State of the art assessment of moisture content (e.g. continuous moisture measurements), structural impact at microscale using CT scanning, NIR tools to verify presence of active components, advanced methods to detect wood rot are all part of a new methodology to predict service life of wood based products in general and in particular as construction component, e.g. new engineered wood products like CLT.

At the Ghent University the Laboratory of Wood Technology has developed a continuous moisture measurement (CMM) set up to assess by means of simulated field testing the time of wetness based on material characteristics. This CMM was first used to check the outstanding performance of plywood in exterior applications often based on non-durable wood species and often without the need to incorporate additional biocides. Also different wood species as well as modified and hydrophobated solid wood have been assessed. Additional laboratory testing was introduced to check moisture dynamics by means of methods including floating or submersion in combination with drying, all in search for correlation with the time of wetness recorded with the CMM equipment.

Key words: *wood products; durability; service life; moisture dynamics; material resistance.*

INTRODUCTION

Wood and wood based products are similarly to the mainly man-made alternatives limited in their ability to remain functional over time. They are inherently prone to biodegradation under natural conditions of the ecosystem cycle and as such all end uses are impacted to some extent. The risk of wood degradation mainly depends on the application conditions. For example, decay fungi are ubiquitous and can grow everywhere as long as the environmental conditions are suitable. The growth of fungi requires wood substances as nutrient source – so they will decrease the strength of wood materials – and a moisture source.

The risk or hazard of a wooden product regarding fungal degradation depends on typical organisms thriving under dry up to continuous wet circumstances, identified as use classes defined as such in EN 335 (2013). Performance related to service life is depending on the durability or material resistance against degrading fungi. Wood or timber species have been classified for natural durability using a service life approach by means of graveyard type field testing. As mentioned before, high variability is a concern and predicting service life for applications out of ground contact leads often to substantial debate (Kutnik 2013, Suttie et al. 2013). To ensure minimum requirements are met, often highly durable species, mainly tropical hardwoods, are selected while adequate performance is feasible with less durable species. Focusing on softwoods it can be stated that the intrinsic or inherent durability is often insufficient and different treating methods (wood preservation, wood protection, wood modification) are used to enhance the material resistance when the products are intended to be used under harsher circumstances. In addition, new wood based products have been developed as well to answer to this need.

To allow the end user to select from a range of alternatives enabling to accomplish fit for purpose criteria defined by limit states, simple classification systems have been developed in the past that are now evolving toward model based approaches combining resistance to biological degradation and moisture dynamics, allowing to aim for an integrated service life assessment approach. Especially in the construction or building sector an improved assessment of service life is certainly welcomed. European standardization within CEN TC 38 on wood durability is working on this topic and test methodology as well as decision support mechanisms are under discussion. Meanwhile also the forum on how to enhance wood properties is working on this renewed concept, e.g. the International Research Group on Wood Protection (IRGWP) and the European Conference on Wood Modification (ECWM). Although the approach to match performance of wood products with material characteristics seems straightforward several additional parameters seem to interact and require additional stochastic modelling related to different commodities, overall and local climate, design, biological hazard, presence and probability of decay organisms, time of wetness, etc.

In this introduction, some elements are provided related to a fit for purpose flow chart for decision making related to parameters defining material characteristics of these wood based products, a selection of state-of-the-art tools available to assist in assessing parameters during exposure and related reliability analysis. Hereafter a short description is given of different use classes, an overview of different wood treatments and innovative wood products that are intended to be assessed within a service life prediction framework. Finally some details are included on the toolbox for non-destructive monitoring of samples exposed in the laboratory or outdoors. Also a brief snapshot of statistical processing of mass loss data from lab testing and time-to-failure analysis of field data, and some specific aspects of service life prediction like benchmarking are presented.

Use classes

The 5 use classes are defined in the EN 335 and ISO 21887 (Suttie et al. 2013) as:

UC1: Situation in which the wood-based product is inside a construction, not exposed to the weather and wetting. Interior, dry.

UC2: Situation in which the wood-based product is under cover and not exposed to the weather (particularly rain and driven rain) but not persistent, wetting can occur. Interior, or under cover, not exposed to the weather. Possibility of water condensation.

UC3: Situation in which the wood-based product is above ground and exposed to the weather (particularly rain). Exterior, above ground, exposed to the weather.

UC4: Situation in which the wood-based product is in direct contact with ground and/or fresh water; Exterior in ground contact and/or fresh water.

UC5: Situation in which the wood-based product is permanently or regularly submerged (i.e. sea water and brackish water). Permanently or regularly submerged in salt water.

Wood preservation

Especially heavy duty applications like transmission poles and railway sleepers have triggered wood preservation. In many countries wood preservation is still considered mainly for use class 4 applications. The assessment of toxic threshold levels for the biocides used is still defined accordingly. Field testing in ground contact combined with a so-called 'no risk' approach is the basis to continue using heavy duty preservatives like creosote, CCA, PCP, etc. However, wood preservation evolved and exterior out of ground applications have become more apparent. When soft rot is of no concern the assessment of wood preservatives in ground contact may lead to excessive requirements for biocides or overkill of recommended or required retentions. Furthermore, in ground contact (or water contact) a continuous supply of water guarantees the development of fungi. Above ground the climatic conditions are far more dominant and can allow to ensure longer service life by means of protection by design and are also impacted by e.g. coatings. Dealing with the interior climate, e.g. use class 2, the risk of getting higher moisture content in wood for a longer period depends not only on the interior climate. Moisture is linked to dampness and is increased when condensation and/or leakage are involved. A critical parameter here is to lower the risk. In practice wood preservation is linked to industry involved in producing, formulating and applying biocides and hence often related to other application areas of such active components.

Wood modification

Over the last decades, additional actors have been coming to the field of enhanced durability. Technologies working under the denominator wood modification claim to enable this without using biocides. The product brand names often are referred to as 'new' wood species and the main focus might well be on increasing other properties like dimensional stability or weathering performance. Besides thermally modified timber (TMT), chemical modification systems are present especially on the European market. These include acetylation and furfurylation. The so-called non-biocidal systems also cover several methodologies to lower the impact of moisture and are known as oil treatments and hydrophobation like the use of organo-silicon compounds. Clearly these methods come close to the role of exterior wood coatings. Currently the industry involved in these technologies seems to be hardly linked to the producers of wood preservatives.

Innovative wood products

On the scene of service life of wood products not only different treatments to enhance durability are of importance. Mainly since mid of last century many innovative wood products have been developed all having an impact on the moisture dynamics and hence on the rate of fungal degradation or service life. For construction, end uses commodities have been developed often intended for protected interior use. Especially under use class 2 the processing of wood into different engineered wood products (EWP) lowers the risk of high humidity over longer periods. Products like glulam (glued laminated timber), CLT (cross-laminated timber), LVL (laminated veneer lumber), LSL (laminated strand lumber), PSL (parallel strand lumber) and other structural composite lumber (SCL) all have different moisture dynamics than solid timber. Furthermore, I-joists and wood I-beams combine several of these and link with the wood based panels (WBP) like plywood and OSB (oriented strand board). Both are often considered for exterior applications and again a major impact of coatings is part of such applications. Coated plywood is an eminent product in this respect. In the group of innovative wood products one should not forget several composite products being developed next to the more traditional ones. With focus on decking and cladding WPC (wood polymer/plastic composites) are clearly stating argumentation of moisture control as one of the main features (Defoirdt et al. 2009). Similarly, natural fibre composites (NFC) based on other 'non-wood' lignocellulosics have similar issues on moisture control to ensure service life in exterior applications (Defoirdt et al. 2017). Unfortunately, often the industries involved in these products have limited links to the companies involved in wood preservation or wood modification.

A framework for service life prediction

The importance of service life prediction for wood and wood based materials is beyond questioning. If proper test methods, monitoring tools and analysis software are tailored to one another, taking into account the specificities of wood, proper service life prediction is at hand. This is not merely a scientific question, the Construction Products Regulation (CPR) on a European level requires reliable information about product performance and is laying down harmonized conditions for the marketing of construction products. Specifically, we aim here at an integrated approach related to service life predication for natural durability, wood preservation, wood modification and innovative wood products with focus on use classes 2 and 3.

The exposure dosage is a measure for the degree of exposure indicating the potential of decay. Fig. 1 shows the macroclimate as defined by the geographical location and climate data for the region together with local impact identified as microclimate will lead to specific time of wetness of the wooden product (Brischke et al. 2010, Rapp et al. 2000). Design will act as an additional parameter lowering or sometimes increasing (e.g. water trapping) the impact of the climate factors. Clearly the climate and especially the microclimate is not or less relevant when considering applications interior dry (UC1) and in soil and (sea) water contact (UC4/5).

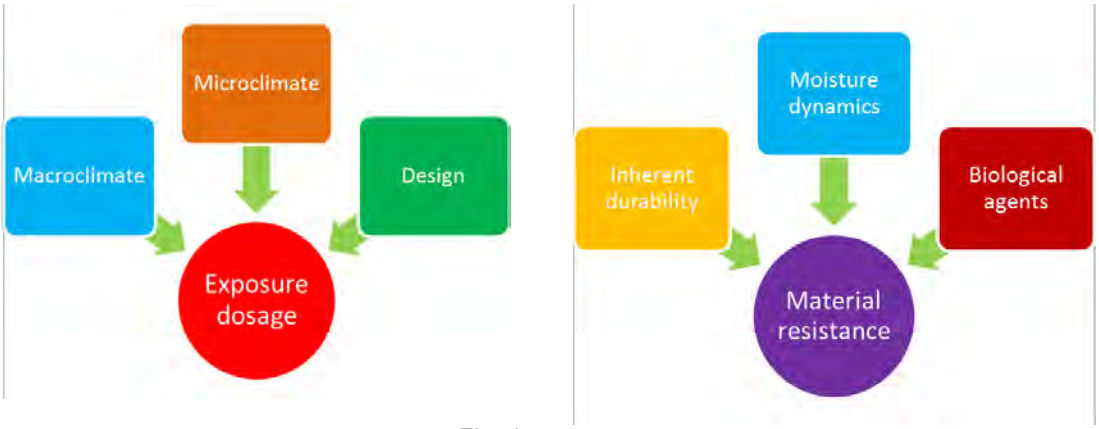

Fig. 1.
(left) Exposure dosage is a combined parameter including macro- and microclimate and design components. (right) Material resistance of wood products is a combination of inherent durability (toxicity), moisture dynamics and these are relevant for the different biological agents.

Any wood species, wood product - treated or not - will have a material resistance against biological agents. Material resistance can be expressed as parameters to assess the degree of resistance related to a biological hazard (Fig. 1). The biological agents of concern differ depending on the use classes and even more to the earlier definition of biological hazard classes. When focusing on fungal decay, the inherent durability can be linked to the presence of biocidal components and hence the level of toxicity towards the fungi of concern. Additionally, it is relevant that the substrate contains a sufficient level of moisture over time. The resistance of any substrate to get wet and the ease or difficulty to dry fast when the wetting stopped can be expressed under the term moisture dynamics (Van den Bulcke et al. 2013, Brischke et al. 2014, Van Acker et al. 2014) and translated into time of wetness (ToW). This parameter is inevitably less relevant when discussing biological agents like drywood insects (HC1) and soft rot / termites / sea organisms (HC4/5).

The parameter interaction defining exposure dosage as presented in Fig. 1 can be presented using 3 axes in a 3D plot for each of them. In Fig. 2 the macroclimate is simply presented as ranging from dry cold to wet warm passing dry warm, continental, maritime and wet cold as intermediate levels. Although this presentation might assume some linear scale the presentation is just to show what components could be considered. Any climate model that can depict the impact on the time of wetness could be used here. When adding the microclimatic impact also some components are listed and the increased impact is not always fully assured. Exposure to the south or north, whether forest cover is present or the exposure is fully open and probably a full sea shore exposure will all have impact mainly on the drying component and limit or increase the time of wetness considerably. Finally, also design qualification will help in identifying different use class levels: (interior) dry, presence of condensation/leakage, out of ground contact without or with water trap and finally contact with soils/water and even sea water. Fig. 2 uses a quasi-continuous approach with outcome levels 0 (blue) up to 5 (red) linked to the harshness of the exposure or simply exposure dosage. The levels 2-3 will probably be most impacted by all variables and need careful concern on how to measure. The different exposure scenarios developed for field testing can be used to verify the parameter time of wetness (ToW). A common approach is actually to work with simulation exposure using semi-field testing (cf. CMM below). This hybrid in service testing also requires adequate statistical approaches as explained further on [8]. When using this approach for a construction commodity, failure can be assessed based on the detection/occurrence and impact of the decay/degradation, but can also be registered based on time of wetness (ToW) determination (Van Acker et al. 2014, Van den Bulcke et al. 2011).

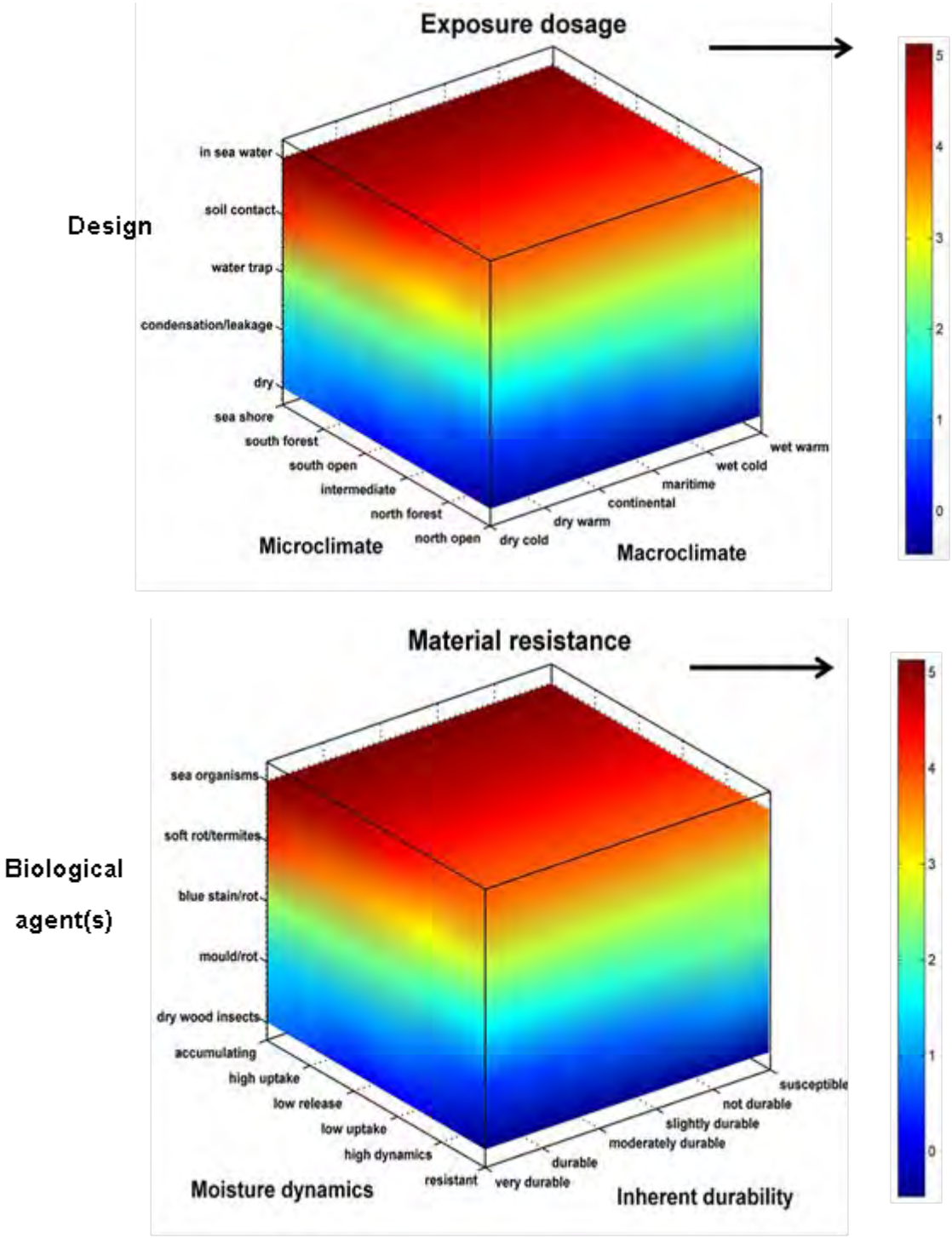

Fig. 2.
(top) Quasi-continuous outline of the exposure dosage (cfr. use classes) in relation to macroclimate, microclimate and design. (bottom) Quasi-continuous outline of the material resistance in relation to inherent durability and moisture dynamics and interacting with biological agents (hazard approach).

Contrary to exposure dosage which is basically independent of the wood product one should assess each commodity intended to perform for a certain time period (service life prediction, SLP). The material resistance is a set of characteristics that determines to what extent one can expect the material to withstand specific biological degradation or even all types of degradation (e.g. weathering). In Fig. 2 the parameters inherent durability, merely due to the presence of active biocidal ingredients, and moisture dynamics are presented in combination with different biological hazards again as a quasi-continuous 3D plot. The material resistance can be checked solely using laboratory testing.

Since different biological agents are acting differently it is necessary to have a multi-agent assessment installed considering that different combinations will be relevant depending on the maximum exposure dosage focusing on. As indicated earlier, when dealing with fungal decay, it is relevant to assess via organism related testing the 'nutritional value' or intrinsic nutritional quality / toxicity. Here again it is important to assess variability and reliability using e.g. life distributions (De Windt et al. 2013). Clearly material using only aspects of moisture control will have some limitations but should anyhow be taken into account when considering increased service life. Here is also an option to link with the performance of coatings.

Linking the in-service assessment of exposure dosage and the lab testing based assessment of material resistance allows to combine both integration and interaction. Integration is mainly linked to the fact that any exposure is somewhere located in the 3D plot as presented in Fig. 2 and that any wooden commodity will have an intrinsic or enhanced material resistance that can be positioned on the 3D plot in Fig. 2. The interaction however is based on what level of material resistance is required for a specific exposure dosage. This many-to-many link requires a (quasi-) discrete approach. For the exposure a dosage range could be determined. Hence a probability or priority of risk could be selected, e.g. the most probable or number one (priority) biological agent. Based on selected laboratory testing one could prioritize results and/or use weighing schemes. Adding input on reliability-based durability for each agent the data on material resistance from lab testing should be used to predict in-service field data. Any qualification of performance of a wood product could use such an early predictor method, but will need to be followed up using full field exposure and assessment of end use performance. This approach has been integrated in the book on Performance of Bio-based Building Materials by Jones and Brischke (2017).

Within the framework to assess the risk of performance failure of wood in outdoor exposures the Time of Wetness (ToW) can be regarded as a tool to illustrate the possibility to degrade or not by fungi (Van den Bulcke et al., 2013). ToW is defined as the time during which a specimen reached a minimal wood moisture content of 20% or 25%. These limits were chosen based on the fact that Hunt and Garatt (1938) considered 20% wood moisture content as the lowest limit, whereas Viitanen (1997) stated that wood moisture content around fibre saturation point (25 – 30%) is required for onset of decay. To allow regular moisture measurements, a continuous moisture measurement (CMM) set up has been installed based on recording of the voltage outputs of calibrated load cells while simultaneously weather data are also monitored. Recorded voltage data are converted to moisture content by using the calibration curve of the load cell and the oven dry weight of the specimens. This CMM was first used by Van den Bulcke et al. (2013, 2009) to check the outstanding performance of plywood in exterior applications often based on non-durable wood species and often without the need to additionally incorporate biocides. The objective is to link specific laboratory immersion tests with outdoor CMM data (Continuous Moisture Measurements - ToW concept). Since both wetting and drying parameters are important a test method was developed using first water permeability (absorption using contact with water during 144h) followed by water vapour permeability (desorption in a second step for 144h). As such lab testing reflects the risk of water accumulation and relates to the ToW measured outdoors. In search for correlation with the time of wetness recorded with the CMM equipment different wood species as well as modified and hydrophobated solid wood have been assessed, however most promising results were obtained for plywood (De Windt et al. 2017).

METHODS AND MATERIALS

Standard lab and field testing and non-destructive monitoring

Standard laboratory based decay tests exist to determine the durability of a wood based material, as well as different field tests. The assessment of such tests is often either expressed in terms of mass loss, and / or in terms of the subjective rating of the condition of the specimen. Given that more objective measures are needed, other techniques, preferentially non-destructive ones, are becoming part of the tools available to assess the condition of a specimen through time. A couple of them will be highlighted briefly here, however many other techniques are not presented here. The data generated by these methods enables to objectively quantify the state of a certain material in function of recorded variables and can be used as input for the statistical framework which will be described further on.

Continuous Moisture Measurement (CMM)

The CMM test set-up consists of a series of single load cells fixed on a table (Van den Bulcke et al. 2009, 2011). On top of the load cells aluminium T-shaped holders are fastened, which are bent at an angle of 45° and on which samples are mounted. The set-up is facing south-southwest to capture maximum rain and solar radiation. This system records the load cell responses with a logging

interval of 5 min. Calibration of the load cells enables to accurately calculate mass changes through time. Adjacent to the CMM set-up a fully equipped weather station is installed consisting of a solar radiation sensor, a tipping bucket rain gauge, a relative humidity probe, a thermometer, an anemometer and a wind vane. As such weather data is collected as well. This set-up enables continuous monitoring of the moisture behavior (ToW) of specimens in outdoor exposure and represents a reliable outdoor (semi field) testing. In combination with local electrical resistance based moisture measurements, this proves to be a very useful method (Li et al. 2013, 2016b).

X-ray Computed Tomography (X-ray CT) scanning

X-ray computed tomography (CT) has proven to be an invaluable technique in various research fields and many types of commercial scanners are available tailored to different needs. The use of X-ray CT in wood research has also increased considerably the last decade, given the significant increase of related publications. X-ray CT as a tool for qualitative and quantitative assessment has become a common tool in wood research with specific focus on micro CT scanning. It allows to visualize the internal structure of an object in a non-destructive way and is as such invaluable for continuous monitoring. It also allows to track, in 3D, moisture absorption and desorption in wood and wood based products (Van den Bulcke et al. 2011, Li et al. 2016a).

Vibration analysis (Resonalyzer)

Flexural vibration has been used to non-destructively measure the mechanical strength of wood based materials (Haines et al. 1996). A so-called 'Resonalyser' technique was further developed to determine the elastic properties of orthotropic plates from resonance frequencies (Lauwagie et al. 2003) of free hanging specimen. These methods are thus feasible to non-destructively visualize and quantify the mechanical strength of wood, but also of wood products such as plywood and MDF (Li et al. 2016).

Near-InfraRed Imaging (NIR)

Investigating wood and wood-based materials using infrared spectroscopy has since long been explored (Schimleck et al. 2002, Kelley et al. 2004, Thumm et al. 2010, Tsuchikawa and Schwanninger 2014). Only point-by-point data could be gathered in the past, yet recently, image-based systems have been developed that are already used frequently in food and pharmaceutical industries for rapid screening (Koehler et al. 2002). Such hyperspectral cameras are able to collect spatial and spectral information simultaneously, resulting in a volume with a spectral profile for each pixel of the image. Obviously, 2D NIR scanning is thus a valuable complementary technique to X-ray CT imaging (Defoirdt et al. 2017).

Statistical and reliability analysis

Mass loss data from decay tests, rating data from field test, and more advanced data generated from abovementioned techniques need to be processed properly. Most importantly, the variability which is present, has to be dealt with statistically. Variability is important on an intra- and inter-product level. Naturally one tries to minimize intra-product variability to maximize inter-product comparability. It is obvious that by implementation of a set of specimens as similar as possible, both the variability of the test itself as well as the material variability is accounted for. A large dataset is said to be a representative selection of a population. Naturally the size of such a set is limited by logistic feasibility, thus one is forced to balance the number of specimens (and sample size) that can be tested with the variability expected and the required certainty. Fitting of probability density functions (pdf) and the implementation of confidence limits as part of reliability analysis should be part of the toolbox to study performance of wood and wood-based products. It has been shown that 2- and 3-parameter Weibull distributions are good candidates. An example is shown in Fig. 3 of mass loss data of different wood species (De Windt et al. 2013) subjected to the lab fungal test CEN/TS 15083-1 (2005).

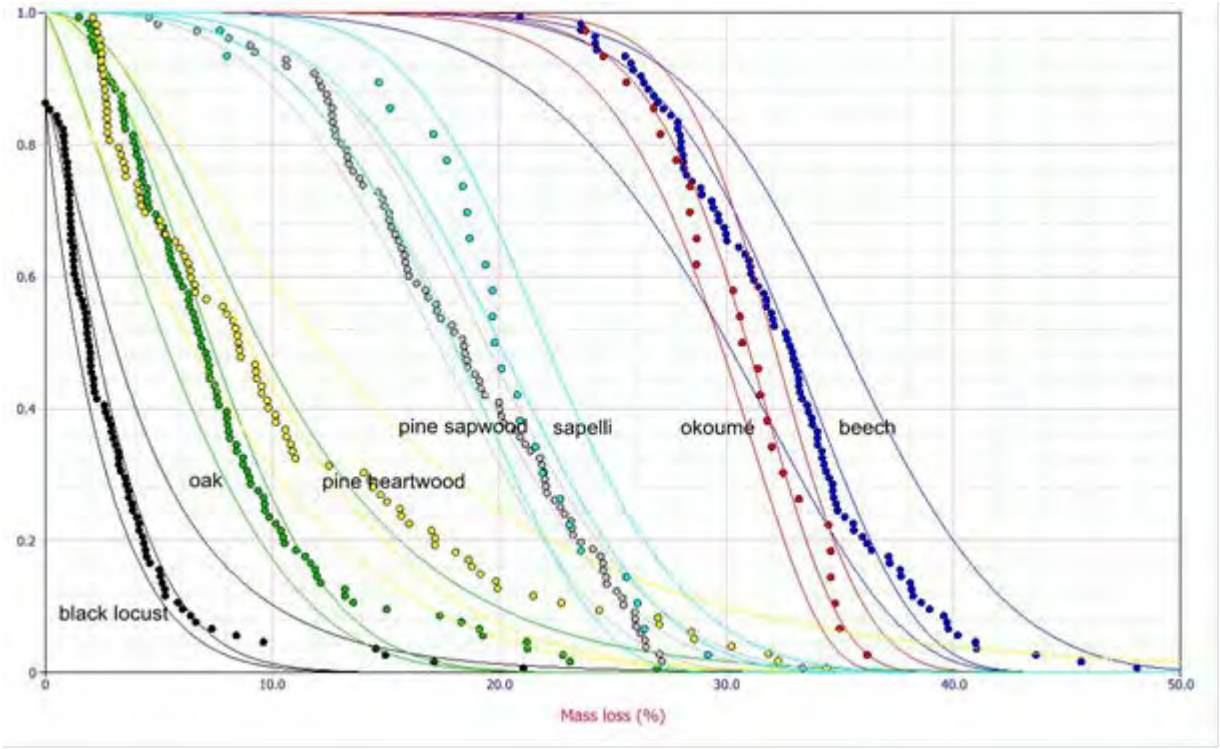

Fig. 3.
Cumulative distribution functions (cdf) for the different wood species with the 95% confidence intervals.

For field testing, at least two extra variability factors have to be considered: probably inhomogeneous conditions on a single field test and the differences between different field sites. Moreover, the factor time and related time-to-failure are crucial in this kind of tests. Even more than in laboratory testing it is obvious that the amount of specimens tested is crucial to get an overall assessment of the different types of variability. This has the implication that even more specimens are necessary to be able to assess performance with a sufficient level of certainty, i.e. increasing confidence of our test results. Therefore, reliability calculations further complicate, since different sites represent an exposure to different stress levels. In such a case it is possible to fit an acceleration model (e.g. an Arrhenius function) to the different distribution functions, i.e. in addition to time-to-failure and the related probability, a factor stress is added to represent the dose-response function we are looking for. As such the complete representation of the probability of time-to-failure for different stress levels will be a three-dimensional function, not only tackling sample variability, but also variability between fields and changes through time.

Benchmarking

Service life predictions methods like the one identified in ISO 15686-8 (2008) use reference service life as a basis for comparison. This approach can be very useful when discussing different parameters related to the fit for purpose objective. Reference products or commodities can be used for benchmarking. Especially when longtime service life appraisal exists for specific wood products it is useful to compare their performance directly with the innovative products under evaluation. Hence it is recommended to include in all types of lab and field testing not only reference products that are deemed to fail or perform higher than expected, but especially the commonly known and accepted products. Clearly the end user would accept and implement innovative products easier if one can prove they are similar or better than the products generally accepted as adequately performing, meaning fit for purpose.

Materials

Softwood as well as hardwood, untreated as well as modified and hydrophobated solid wood were tested. An overview of all materials under test was given in Table 1.

Table 1

Overview of the test material

Softwoods			
Code	**Wood species**	**Key botanical species**	**Modification - Treatment**
RAD PINE	radiata pine	*Pinus radiata* D.Don	-
PINE-SAP	Scots pine (sapwood)	*Pinus sylvestris* L.	-
PINE-HW	Scots pine (heartwood)	*Pinus sylvestris* L.	-
SPRUCE	Norway spruce	*Picea abies* (L.) H.Karst.	-
LARCH	European larch	*Larix decidua* Mill.	-
SIB LARCH 2	Dahurian larch	*Larix gmelinii* (Rupr.) Kuzen.	-
W RED CEDAR	western red cedar	*Thuja plicata* Donn ex D.Don	-
TM-RAD PINE	radiata pine	*Pinus radiata* D.Don	Thermal
TM-RAD PINE 2	radiata pine	*Pinus radiata* D.Don	Thermal
FU-RAD PINE	radiata pine	*Pinus radiata* D.Don	Furfurylation
AC-RAD PINE	radiata pine	*Pinus radiata* D.Don	Acetylation
TM-PINE	Scots pine	*Pinus sylvestris* L.	Thermal
TM-SPRUCE	Norway spruce	*Picea abies* (L.) H.Karst.	Thermal
TM-SPRUCE 3	Norway spruce	*Picea abies* (L.) H.Karst.	Thermal
TM-SPRUCE 4	Norway spruce	*Picea abies* (L.) H.Karst.	Thermal
HF-PINE-SAP A	Scots pine (sapwood)	*Pinus sylvestris* L.	Non-ionic emulsion (silane, silicone resin and siloxane)
HF-PINE-SAP B	Scots pine (sapwood)	*Pinus sylvestris* L.	Cationic emulsion (siloxane)
HF-PINE-SAP C	Scots pine (sapwood)	*Pinus sylvestris* L.	Siliconate
Hardwood			
Code	**Wood species**	**Key botanical species**	**Modification**
POPLAR	poplar	*Populus* × *canadensis* Moench cv 'Ghoy'	-
BEECH	European beech	*Fagus sylvatica* L.	-
SAPELLI	sapelli	*Entandrophragma cylindricum* Sprague	-
OAK	European oak	*Quercus robur* L. & *Quercus petraea* (Matt.) Liebl.	-
TEAK	teak	*Tectona grandis* L.f.	-
TM-POPLAR	poplar	*Populus* × *canadensis* Moench	Thermal
TM-BEECH 2	European beech	*Fagus sylvatica* L.	Thermal
TM-OBECHE	obeche	*Triplochiton scleroxylon* K.Schum.	Thermal
TM-LIMBA	limba	*Terminalia superba* Engl. & Diels	Thermal
TM-ASH	European ash	*Fraxinus excelsior* L.	Thermal
TM-CELTIS	celtis d'Afrique	*Celtis adolfi-friderici* Engl. & *Celtis tessmannii* Rendle	Thermal

Hydrophobation was performed by vacuum impregnation of Scots pine sapwood specimens using water based treating solutions containing 10 % active ingredient provided by Dow Corning. The test specimens had cross sections of 50 by 25 mm² and a growth ring angle close to 45°, similar to the stakes as provided for e.g. EN 252 (2014) in ground field testing. The sampling procedure for the results presented in this paper focussed on having matched samples for all tests. At least three replicates of each wood species or treatment were tested.

Laboratory testing of moisture dynamics

Regarding Time of Wetness both surface and end-grain phenomena have an impact on water uptake. Therefore, the earlier developed floating test method by Rapp et al. (2000) to reveal how fast water enters through a wood surface and how easy it dries afterwards was refined and complemented by a submersion test. Both methods are briefly outlined in Table 2.

Table 2

Brief overview of floating and submersion tests to assess moisture dynamics

Parameter	Floating test	Submersion test
specimen cross section	50 by 25 mm²	
specimen length	50 mm	150 mm
edge sealing	yes	no
water penetration	one surface	whole specimens
absorption phase	1, 4, 8, 24, 48, 72, and 144 h	
desorption phase	1, 4, 8, 24, 48, 72, and 144 h	
preferred unit	g/m²	kg/m³

To compare the mass changes of the test materials during water uptake a curve was fitted to the data. The absorption curves fitted for both the floating test and the submersion test are based on the formula equation (1):

$$f(x) = a * x^b \quad (1)$$

When parameter b is close to 0.5, parameter a approximates the absorption coefficient. This is based on the linear relation between water uptake and the square root of time (2002). Deviating b-values indicate special absorption phenomena e.g. capillary water uptake.

Water vapour release show curves based on formula (2):

$$f(x) = a + be^{\frac{-x}{c}} \quad (2)$$

In this equation parameter a approximates the remaining water after drying. Parameter b reflects the amount of water evaporated during 144h desorption. Consequently, the sum of parameter a and parameter b equals the increase in g/m² during 144h absorption. Parameter c is a measure for the desorption rate. A low c-value indicates that the specimen will dry quickly, a high c-value reflects slow drying of the test specimen. In general, high measures for parameters a, b and c indicate an increased risk of moisture accumulation.

Field testing of moisture dynamics

The continuous moisture measurement (CMM) set up aims at monitoring water absorption and desorption behaviour of specimens while subjected to outdoor exposure. The test consists of a frame upon which two parallel series of single load cells are fixed. The precision of the load cells is 1.0 g. All specimens were inclined 45° facing south west direction. Weather data are recorded by means of a weather station consisting of a pyranometer, a pluviometer, a relative humidity probe, a thermometer, an anemometer and a windvane adjacent the test set up. All data were registered by a delta-T logging unit every 5 min. More details concerning CMM can be found in Van Acker and De Smet (2007) and in Van den Bulcke et al. (2009). The continuous moisture measurement (CMM) set up included the same set of material as detailed in Table 1. Specimens were identical to the ones used for the submersion test and cross sections were sealed (Fig. 4). After installation specific rain events were selected to analyse major differences. All data were converted to hourly data by averaging and missing data were omitted from the analysis.

Fig. 4.
Continuous Moisture Measurement.

RESULTS AND DISCUSSION

Laboratory testing of moisture dynamics

Although different absorption and desorption mechanisms were expected resulting in short term (< 1 hour) and long term (> 6 days) results, the results presented here focus on the 144 h absorption and 144 h desorption. Van Acker and co-authors (2014) already ranked a broad range of indigenous and tropical wood species both for the floating and submersion test based on mean values of 144 h absorption and desorption. Values after 144 hours of floating were considered high when over 5000 g/m² and corresponding values for desorption should then be over 2000 g/m². Furthermore, four groups were defined and within each group two subgroups were distinguished as detailed in Table 3.

Table 3

Classification of absorption and desorption for both floating [g/m²] and submersion (kg/m³) based on upper limit criteria.

Class	Floating test [g/m²]		Submersion test [kg/m³]	
	Absorption	Desorption	Absorption	Desorption
1	750	250	90	15
2	950	400	110	20
3	1150	500	130	25
4	1350	600	150	30
5	1750	750	170	40
6	2750	1000	210	55
7	5000	2000	250	70
8	∞	∞	∞	∞

Given the criteria in Table 3 and the parameters derived from curve fitting discrimination between species based on moisture dynamics is possible.

A classification of the test materials along their moisture dynamics is given in Table 4 (softwoods) and Table 5 (hardwoods).

Table 4

Moisture dynamics parameters of softwoods and modified softwoods using classes as defined in Table 3 for floating [g/m²] and submersion [kg/m³]

Softwoods	Floating - absorption				Floating - desorption					Submersion - absorption				Submersion - desorption				
	Class	[g/m²]	f(x)=a*x^b		Class	[g/m²]	f(x)=a+b*exp(-x/c)			Class	[kg/m³]	f(x)=a*x^b		Class	[kg/m³]	f(x)=a+b*exp(-x/c)		
		144h	a	b		144h	a	b	c		144h	a	b		144h	a	b	c
RAD PINE	8	6729	2152	0.22	8	2741	-	6721	174	6	209	79	0.19	6	44	-	210	93
PINE-SAP	7	4256	516	0.42	7	1423	1214	3051	49	7	235	49	0.31	6	45	31	205	47
PINE-HW	4	1259	71	0.58	4	559	592	648	14	5	170	19	0.44	5	32	30	137	37
SPRUCE	5	1503	98	0.55	5	743	786	678	17	5	165	20	0.42	5	36	32	131	38
LARCH	5	1673	112	0.54	5	650	648	935	50	6	185	11	0.57	6	41	-	187	102
SIB LARCH 2	5	1505	93	0.56	5	629	631	783	48	5	169	10	0.57	6	42	11	156	90
W RED CEDAR	4	1314	85	0.55	3	401	387	881	43	4	138	20	0.39	1	12	2	135	54
TM-RAD PINE	8	5715	816	0.41	8	3705	1896	3688	206	7	248	35	0.40	8	72	36	212	84
TM-RAD PINE 2	8	6938	1666	0.30	8	4144	-	6883	300	8	383	78	0.32	8	117	20	364	109
FU-RAD PINE	7	2918	680	0.29	7	1246	-	2844	177	8	258	61	0.29	8	90	-	259	149
AC-RAD PINE	7	3355	713	0.32	7	1923	800	2463	188	6	184	27	0.39	8	69	59	124	61
TM-PINE	1	245	13	0.58	1	99	111	130	4	1	51	2	0.64	1	10	11	40	17
TM-SPRUCE	1	612	30	0.61	1	219	220	390	8	3	111	7	0.55	2	19	20	88	30
TM-SPRUCE 3	1	319	17	0.58	1	112	129	181	3	2	102	11	0.45	2	19	19	81	39
TM-SPRUCE 4	1	600	29	0.61	1	243	265	319	9	1	89	7	0.51	2	17	17	70	36
HF-PINE-SAP A	8	8630	1319	0.39	6	1400	568	8087	63									
HF-PINE-SAP B	8	6793	811	0.43	4	608	364	6436	44									
HF-PINE-SAP C	8	10839	1815	0.38	1	130	-	10995	32									

Table 5

Moisture dynamics parameters of hardwoods and modified hardwoods using classes as defined in Table 3 for floating (g/m²) and submersion (kg/m³)

Hardwoods	Floating - absorption				Floating - desorption					Submersion - absorption				Submersion - desorption				
	Class	g/m²	f(x)=a*x^b		Class	g/m²	f(x)=a+b*exp(-x/c)			Class	kg/m³	f(x)=a*x^b		Class	kg/m³	f(x)=a+b*exp(-x/c)		
		144h	a	b		144h	a	b	c		144h	a	b		144h	a	b	c
POPLAR	6	2210	135	0.56	4	534	523	1677	29	8	264	21	0.51	6	55	30	232	63
BEECH	6	2588	143	0.58	7	1090	1112	1460	27	8	324	39	0.43	8	107	68	257	75
SAPELLI	4	1331	64	0.61	5	735	779	532	9	4	136	9	0.55	5	40	42	93	24
OAK	5	1459	105	0.53	5	687	707	730	19	5	167	15	0.48	5	40	38	127	37
TEAK	1	653	40	0.56	2	263	315	307	9	2	98	5	0.59	3	24	21	77	45
TM-POPLAR	1	557	16	0.72	1	110	135	382	10	3	111	27	0.24	1	14	12	99	36
TM-BEECH 2	3	992	51	0.59	2	348	374	568	21	5	160	14	0.50	5	37	32	128	44
TM-OBECHE	5	1392	50	0.67	2	294	104	1244	75	4	133	4	0.70	1	14	-	133	61
TM-LIMBA	2	768	31	0.64	2	291	305	440	13	5	155			5	31	25	129	50
TM-ASH	3	1099	71	0.55	2	303	328	695	35	4	134	13	0.47	4	27	27	106	39
TM-CELTIS	3	960	45	0.61	3	427	443	484	16	4	132	6	0.63	5	33	29	100	51

Focussing on softwoods first and on pine in particular one can observe the highest uptake combined with a moderate drying rate for Scots pine sapwood. Radiata pine even has a lower absorption amount (but higher rate, parameter a) combined with a faster drying (desorption parameter c). European and Siberian larch are actually rather similar in behaviour when submersion is considered (open end-grain, cross section uptake). Spruce and Scots pine heartwood show a similar rather low uptake as Siberian larch but are slower in drying after submersion. Western red cedar is not only the lowest in absorption it also dries rather fast. All these observations mainly have an impact when wood is exposed to end grain water trapping.

Moisture dynamics along tangential face simulating wetting of a surface during a rain event, revealed the very fast drying of radiate pine and modified radiate pine which is inevitably related to drying after a rain event on an exposed surface. This somewhat special characteristic of radiate pine was also noticed by the absorption b parameter being different from the general 0.5 value. Overall high water uptake is balanced by fast drying. This is not valid for all the modified radiata pine. Thermal treatment and to a lesser extent furfurylation and acetylation had a less beneficial effect on the moisture dynamics of radiate pine resulting in a less favourable classification. Regarding the good durability of modified radiate pine, the wetting ability and drying rate were not representative for its overall material resistance. The set of modified Scots pine and spruce all show a very low uptake (absorption), but also rather low drying rate (c- values) resulting in low ToW.

Pine sapwood treated with the hydrophobation product A showed altered moisture dynamics in comparison to untreated Scots pine sapwood although less altered than treated with product B. For pine sapwood treated with product C an increased absorption was observed. The desorption values were in line with the absorption values indicating low risk for accumulating water leading to issues on ToW. The low c-value for product C however indicated fast drying.

In Table 5 the same parameters were provided for hardwoods. Even more than western red cedar as softwood, teak can be considered as an eminent wood species related to moisture dynamics with limited absorption and higher class for drying than for wetting. Comparing the data and parameters of the modified hardwoods with the TMT spruce and Scots pine (Table 4) only the thermally modified poplar (TM-POPLAR) was showing good moisture dynamics. The values for TM-POPLAR were similar to the ones determined for teak. Sapelli also shows similarity to teak, a species where the desorption class was higher than the absorption class, pointing at a low risk for moisture accumulation. Only the modified celtis allowed for a similar observation though only for the submersion test. Differentiation was higher when considering the submersion test. Quite some of the modified materials showed higher classification on desorption than on absorption, also radiate pine. Ease of drying after getting wet might be a more important parameter than just the ability of getting wet.

Continuous Moisture Measurement (CMM)

When assessing time of wetness related properties moisture content changes of individual specimens were recorded just before and after a rain event during outdoor exposure alongside the actual precipitation. A moderate rain is considered to be between 2.5 and 7.6 mm/h. Details for softwoods are presented in Fig. 5 and 6 and for hardwoods in Fig. 7.

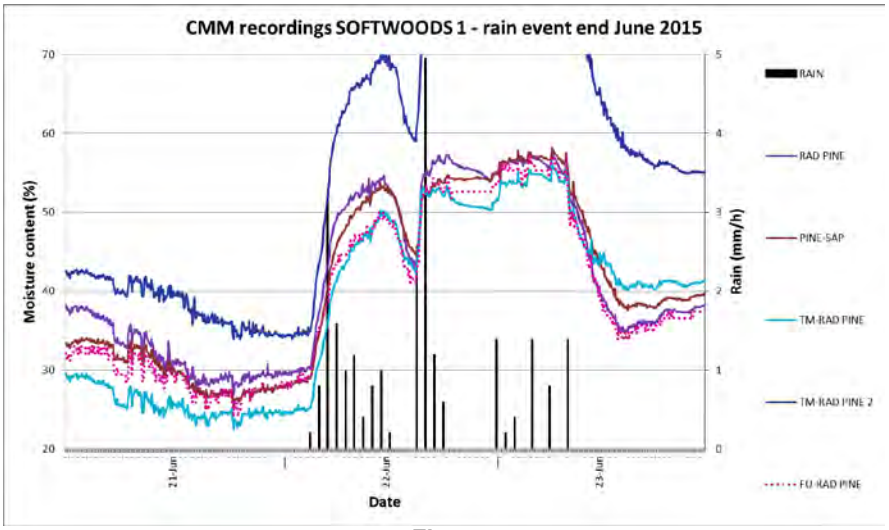

Fig. 5.
Field test moisture recordings (CMM) of softwoods with high absorption.

The wood species with a higher water absorption are presented in Fig. 5. These results confirmed the findings of laboratory testing: radiate pine, thermally modified and furfurylated radiata pine and Scots pine sapwood showed high moisture contents, even higher than 50 % during a rain shower.

The remaining softwoods presented in Fig. 6 had distinctly lower moisture content levels. This group was divided in two subgroups. The first subgroup comprises western red cedar, larch species, Scots pine heartwood and spruce. These softwoods had moisture contents around 20% during dryer periods and moisture contents of maximum 40% during rain events. A second group consisted of wood species with a moisture content of ± 10% during long dry periods and a moisture content up to 30% when it rained. TM Scots pine, acetylated radiate pine and TM spruce (with somewhat higher dynamics) belong to this group.

The hardwoods as shown in Fig.7 were in general overlapping with the above groupings identified for softwoods. Unmodified poplar showed a similar moisture content level as spruce unmodified. Both beech and sapelli had an equally low moisture content during the dry periods with clearly lower absorption during actual rain of sapelli. Oak showed a merely equally positive pattern as sapelli, though more distinct differences between the dry period and during the rain event. Thermally modified TM obeche was more impacted by wetting and drying than TM ash. TM poplar, TM limba, TM beech and TM celtis in this order were showing lower absorption. However, they all came to a similarly low moisture content after this rain event.

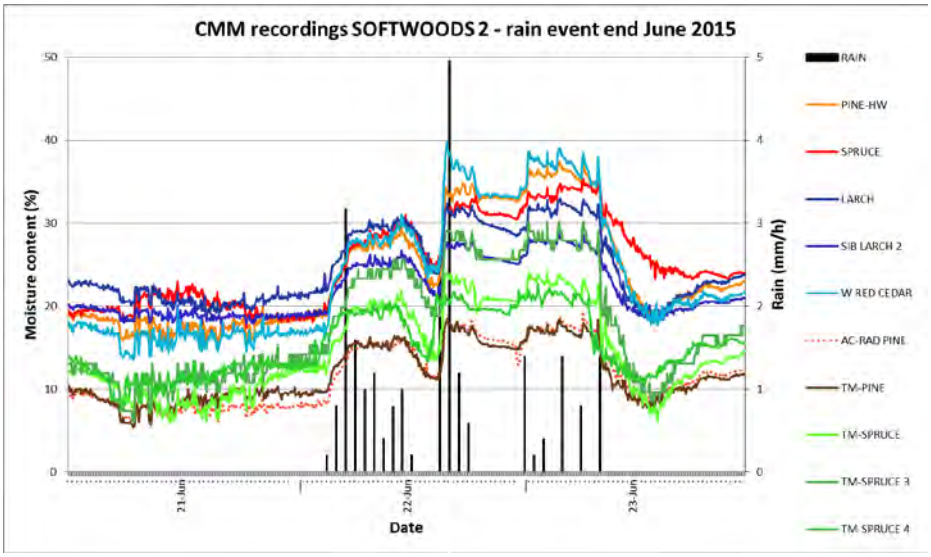

Fig. 6
Field test moisture recordings (CMM) of softwoods.

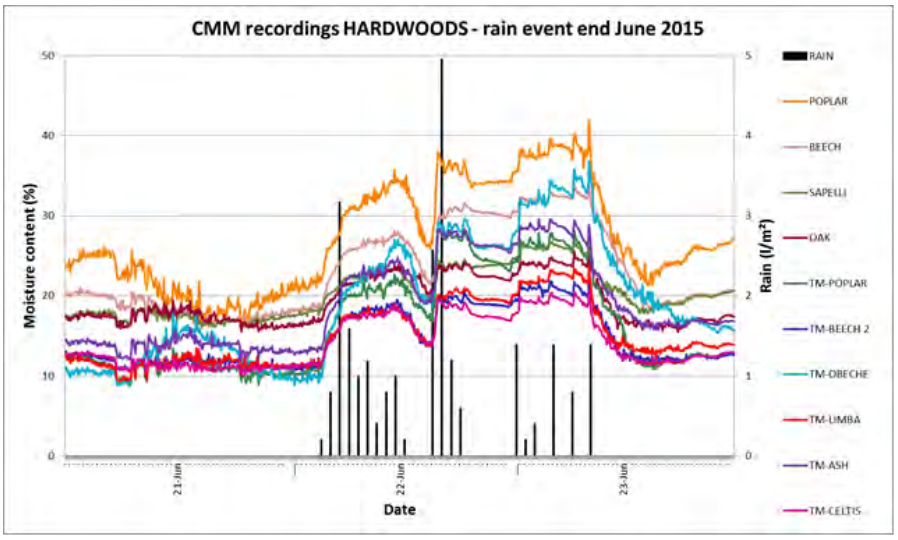

Fig. 7.
Field test moisture recordings (CMM) of hardwoods.

Considering hydrophobing agents all treated Scots pine sapwood treated showed lower moisture content than its reference (Fig. 8).

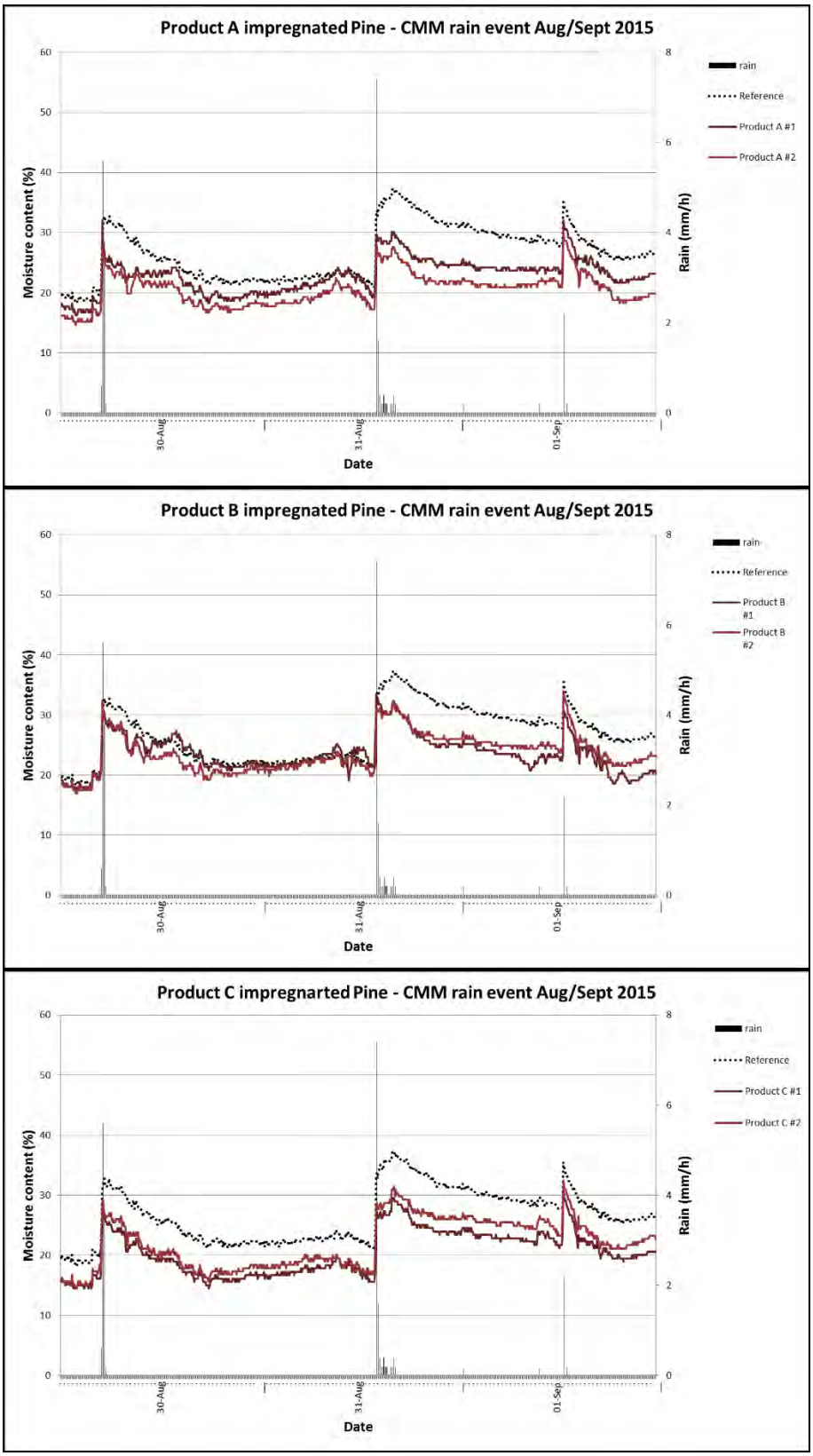

Fig. 8.
The continuous moisture measurement of Scots pine sapwood treated with hydrophobing products A, B and C.

The major differences between the performance of each product was found in the reactivity to rain and the subsequent drying phase. Product C followed the same trend as the reference though at lower moisture content. Product B treated wood did not differ from the reference for the rain shower on August 30th, but was not that sensitive to the longer rain event the next day. The product A induced lower absorption overall but also revealed slower drying. Comparing results from the floating test (Table 4) with the results obtained from CMM is difficult when interpreting small differences between the hydrophobation treatment. Clearly this interrelationship will also change when weathering is more involved.

In general, based on the absorption rate and amount of water uptake as well as the drying rate as derived from laboratory testing of moisture dynamics a good estimate of the risk for moisture accumulation could be made and hence the duration that a material has a beneficial moisture content for the development of rot (ToW) as observed in the CMM.

CONCLUSIONS

The concept of service life prediction (SLP) is of major importance for the utilisation of wood and wood products. Many products are compared to alternatives based on man-made materials. Besides the ability to predict performance it is also relevant to know what is actually expected from a specific commodity. The service life prediction framework presented in this paper including both material resistance as well as moisture dynamics, a toolbox for non-destructive monitoring, decay testing, reliability analysis, and benchmarking can help in selecting fit-for-purpose wood based products in the construction sector.

Material resistance against decay depends partially on the presence of active ingredients preventing or slowing down wood degradation, and partially on the moisture dynamics of the material. The moisture dynamics of indigenous and tropical wood next to modified and hydrophobated wood was studied by means of simple laboratory tests such as the floating test or submersion test. Although both test methods reveal overall similarity the floating test focusses on face water penetration valid for plank surfaces and most wood based panels whereas the submersion test mainly relates to water trapping in constructions. The parameters derived allowed for additional information on absorption and desorption rate, maximal absorption and residual moisture content after desorption.

A continuous moisture measurement during a rain event confirmed this methodology and allowed to discuss the moisture dynamics by means of time of wetness (ToW, time of being above a certain moisture content). Interesting species related differences reflecting practical experience were found. However, the importance of the moisture dynamics can be overruled by the intrinsic natural durability of wood or the enhanced durability by means of special treatments e.g. wood preservation or wood modification. This was clearly illustrated by the increased ToW of modified wood suggesting an increased risk of decay although modified wood is durable.

Linking service life and moisture dynamics is not straightforward, neither is durability testing sufficient to show full potential. Nonetheless, the ToW is a valuable measure in the performance assessment of wood and wood based products.

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ALTERNATIVE RAW MATERIALS FOR BIO-BASED COMPOSITES

Petr KLIMEK
TESCAN Orsay Holding, a.s., Global Applications & Material Science,
Brno, Czech Republic
E-mail: klimekpeta@gmail.com

Rupert WIMMER
University of Natural Resources and Life Sciences, Vienna
IFA-Tulln, Institute for Natural Materials Technology
Konrad Lorenz Strasse 20, 3430 Tulln, Austria
E-mail: Rupert.Wimmer@boku.ac.at

Abstract

This contribution is a state-of-the-art review and synthesis on alternative materials used for bio-based composites, in particular for particleboards. Basic economical and ecological aspects of wood replacements for particleboards are discussed, and effects of on various mechanical properties are compiled. Synthesis and Ashby plots provide information useful for the optimization of composite materials. Is is shown that alternative materials are available at sufficient volumes, at potentially lower prices, and with benefits such as termite resistance, or reduced swelling rates. However, mechanical properties are mostly lower, when compared to wood-based particleboards. It can be stated, that bio-based composites, i.e. particleboards, made from alternative materials, widely show a satisfying overall performance at lower prices.

Key words: *Bio-composite; particleboard; mechanical proeprties; composites; waste; cleaner production; manufacturing; resource efficiency; agriculture; residues.*

INTRODUCTION

Composite materials can be defined as a combination of two or more materials that results in better properties than those of the individual components used alone. The material components retain their separate chemical, physical, and mechanical properties, and the (at least) two constituents are called matrix and reinforcement (Campbell 2010). A composite could be either dominated by the matrix, or by the reinforcement material, resulting in matrix-dominated and fiber/particle-dominated composites, respectively. In most cases, the reinforcement is harder, stronger, and stiffer than the matrix, which rules the obtained properties. There are also materials that can be positions between the two types, such as as thermoplast-bonded wood-based panels, moulded chipboards with a higher proportion of adhesives, or non-woven materials (Figure 1).

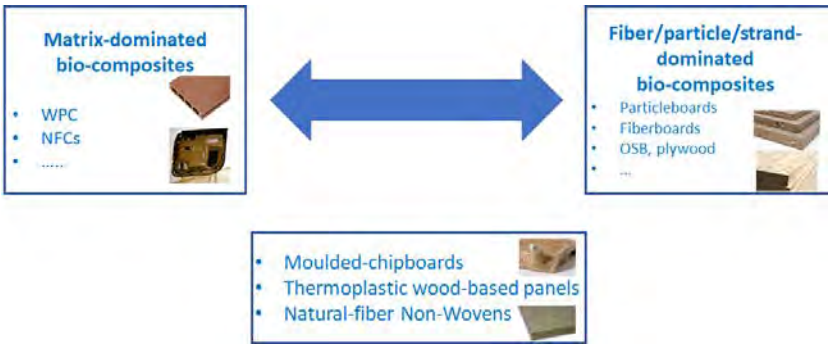

Fig. 1.
Matrix-dominated vs. fiber/particle/strand-dominated bio-composites, and materials positioned in-between.

Particleboards as a particle-dominated bio-composite have been around for more than 120 years. They were initially developed to utilize wood wastes coming from sawmills, and other processing sites, to be used as a low-cost alternative to panels such as plywood, or blockboards ("*Tischlerplatte*"). Particleboards hold the strongest market position, representing over 60% of the

entire wood-based panel production in the European Union. The total annual volume of particleboards produced in Europe equals to 28.4 million m^3 (EPF, 2014). Today, the majority of the raw material used for particleboards (~70 %) is low-grade virgin wood. However, low-grade timber is used for cross-laminated timber, or for glued-laminated timber ("Glulam"). The high demand of low-grade timber, along with termporary supply shortages are causing cost increases for wood chips (Eastin et al. 2012). Prices for wood chips have increased up to 30% between 2006 and 2011. Increased wood resource prices may cause unstable particleboard supplies, with the consequence of higher market prices and reduced competitiveness. As a response, strategies to achieve a higher resource efficiency have been initiated (European Commission 2011; Fischer-Kowalski et al. 2013). The goal here is to achieve a cleaner and more environmental friendly production, and also sustain the European resource situation. The currently increased raw material prices may also motivate companies to improve productivity, and invest also in resource-efficient technologies (Giljum et al. 2009). Suggestions for particleboards produced with alterative resources are welcomed politically, and also economically. In this contribution, particleboards as the most prominent wood-based panel are reviewed with respect to alterative raw materals to be utilized in their manufacturing.

MATERIAL AND METHOD

A state-of-the-art literature review was done on alternative raw materials used in particleboard manufacturing. The entire review covers aspects such as (1) availability of alternative materials with respect to market prices and volumes, (2) alternative particleboard developments using straw, plant stalks, prunings, other natural materials, and also non-plant wastes, and (3) material selection criteria, including so called Ashby plots. Ashby (1992) suggested material property charts, by plotting one property against another, obtaining maps with property-spaces that can be identified. The obtained regions are then usually occupied by different material classes. These plots help to suggest alternative materials suitable to replace wood, with properties to be optimized.

RESULTS AND DISCUSSION

Market availability

Market prices for materials obtained from agricultural resources, along with the applicability of common econometric tools, are widely lacking. There is a lack of knowledge on production capacities, with insufficient information on potential markets and market prices. Prices are highly dependent on local supplier volumes, and harvesting costs, the latter including chopping, baling and on-farm hauling of crops, among baling to be considered as the most expensive step (Khachatryan et al. 2009). A central aspect that may turn higher prices to lower ones is seen when an utilization of wastes and residues is considered (Gallagher et al., 2003). It should be kept in mind that many residues are currently utilized for different purposes, such as for cattle feeding, soil erosion control, or soil nutrition, with the consequence that final prices are strongly determined by the current demands. Price estimates for materials potentially suitable for particleboard production are compiled and listed in Table 1.

Prices for wood chips in Europe are reported to be 59–65 €/t (as of 2014). Lesser used crop residues coming from agricultural plants such as corn stover (stalks and leaves), wheat straw, or plant stalks, may offer options for substantial particleboard production savings. Estimated prices for these plant residues are at least 50% below the wood chips prices (Table 1). However, as prices are valid for certain regions only, they are subject to high variability, and depenedent on transportation costs and the cultivated volumes. However, by assuming long-term supply contracts, prices for crop residues are estimated to be at 40 €/t (2014, Table 1).

Wood chips are ranked second within the production cost structure, behind the first-ranked resin costs. For medium-sized particleboard plants (production < 140 000 m^3/year), prices for wood chips may be as high as 4 Mio €/anno, which represents 20% of the entire cost structure for a given particleboard manufacturer. Thus, replacing wood by cheaper materials may provide substantial production cost savings.

Table 1

Market prices for selected raw materials

Material type	Prices	
Crops residues [US]	**17 – 32 €/ t** (Sokhansanj & Fenton 2006; Khachatryan et al. 2009)	**13 – 27 €/ t** (Gallagher et al. 2003)
Wheat straw [US]	**24 – 35 €/ t** (Perlack et al. 2011)	**19 €/ t** (Gallagher et al. 2003)
Rice straw [US]	**23 €/ t** (Gallagher et al. 2003)	**6 -37 €/ t** (Jenkins et al. 2000)
Corn residues [US]	**13 €/ t** (Gallagher et al. 2003)	
Wood chips [EU]	**59 – 65 €/t** (Malins et al. 2014)	**65 €/t** (Spelter et al. 2008)

A medium-sized particleboard plant requires e.g. 106 000 tons (dry mass) of wooden resources, to produce 140 000 m^3 particleboards (Spelter et al. 2008). In a nutshell: to produce 1 m^3 of particleboards, 0.75 t of raw materials are required. The annual particleboard production in Europa of 28.4 million m^3 is therefore equaling to an approximated consumption of 21 Mio tons of wood. To convert that biomass to forest land-use, assuming 10 $t \cdot ha^{-1} \cdot year^{-1}$ of biomass production (Pretzsch, 2009), a total of 2.5 Mio ha forest land is needed to secure the raw materials required for one year of wood-based particleboard production. If non-wood resources are seen as an option, they don't have to be necessarily cheaper, but they should show a similar biomass yield, and also sufficient land availability.

Yield for agriculture plants may be similar to higher, compared to biomasses obtained from forests (Table 2). Yields with up to three times higher than forests are obtained with Giant Miscanthus (*Miscanthus x giganteus*), demonstrating a biomass production at 44 $t \cdot ha^{-1} \cdot year^{-1}$ (Pyter et al. 2007). Miscanthus is becoming more and more popular in the colder, northern European climates (Monti et al. 2015; Parajuli et al. 2015) as a bioenergy crop (Ameline et al. 2015), or is used as a resource in chemical production (Arnoult et al. 2015; Kim et al. 2015). Higher yields than wood are also demonstrated by cup-plants (*Silphium perfolatium L.*), reaching 40 $t \cdot ha^{-1} \cdot year^{-1}$. This plant originates from Eastern North America (Stanford 1990), but is now widely established across Central Europe. This plant has been cultivated in gardens as an ornamental plant during the 18^{th} century, and it is nowadays widely planted for energy purposes (Haag et al. 2015). Aspects of cultivation and utilization of cup-plant is reviewed by Gansberger et al. (2015). Plantations of these plants may be done on lesser-used, low-productive lands. Currently, 24 Mio ha of land in Europe is not in-use. It can be assumed that 3% of European unused land could be cultivated with cup plant or Miscanthus, which would be sufficient to secure the raw material demand for the entire European particleboard production.

Sunflower stalks are obtained from fields intentioned for sunflower oil seed production, and the same is true for rapeseed (*Brassica napus L.*) stalks. In Europe, sunflowers are occupying 4.25 Mio ha, while rapeseed plants are cultivated on 6.6 Mio ha (Krautgartner et al. 2016). Sunflower stalks may potentially replace up to 80% of the wood used in European particleboard production, while rapeseed stalks may provide 41 Mio tons per year, which is twice the amount needed for the entire European particleboard production.

Table 0

Biomass yields for selected raw materials

Material	Yield	
Forest biomass	**5.9 t·ha^{-1}·year^{-1}** (Amaro et al., 2003)	**6 - 16 t ha^{-1}·year^{-1}** (Pretzsch, 2009)
Pulp wood	**2 – 10 t ha^{-1}·year^{-1}** (Campinhos, 1999)	
Spruce wood	**6.1 t ha^{-1}·year^{-1}** (Campinhos, 1999)	
Topinambour stalks	**6 - 8 t ha^{-1}·year^{-1}** (Dix et al., 2009)	**8 t ha^{-1}·year^{-1}** (Meyer, 2007)
Hemp	**6 – 7 t ha^{-1}·year^{-1}** (Dix et al., 2009)	
Rapeseed stalks	**6.3 t ha^{-1}·year^{-1}** (Dix et al., 2009)	
Wheat straw	**3 - 5.8 t ha^{-1}·year^{-1}** (Dix et al., 2009)	
Sunflower	**3.9 t ha^{-1}·year^{-1}** (Dix et al., 2009)	
Maize stalks	**8 – 15 t ha^{-1}·year^{-1}** (Dix et al., 2009)	
Cup-plant	**7 – 16 t ha^{-1}·year^{-1}** (Gansberger et al., 2015)	**40 t ha^{-1}·year^{-1}** (Stanford, 1990)
Miscanthus	**23 - 44 t ha^{-1}·year^{-1}** (Pyter et al., 2007)	**12 - 20 t ha^{-1}·year^{-1}** (Monti et al., 2015)

Wheat straw is grown in the EU at a mass that is three-times (74 Mio tons per year) the biomass needed by the particleboard industry. However, wheat straw is also used for the production of energy through combustion (Kretschmer et al. 2012). Topinambour stalks are produced at about 600 000 tons / year (Izsáki and Kádi 2013), which could replace 3% of the wood used for particleboards in Europe. Hemp stalks are available at amounts of about 105 000 tons (Carus et al., 2013) per year. Hemp is also used as an insulation material, in polymer-based composites, for non-wovens, or for making paper. Maize is taking 15 Mio hectares of land, with 60% harvested for grains, and 40 % for silage (ESA, 2016). This volume exceeds six times the raw material demand needed for EU-produced particleboards. Availability biomass of wood and non-wood raw materials for particleboards are summarized in Figure 2.

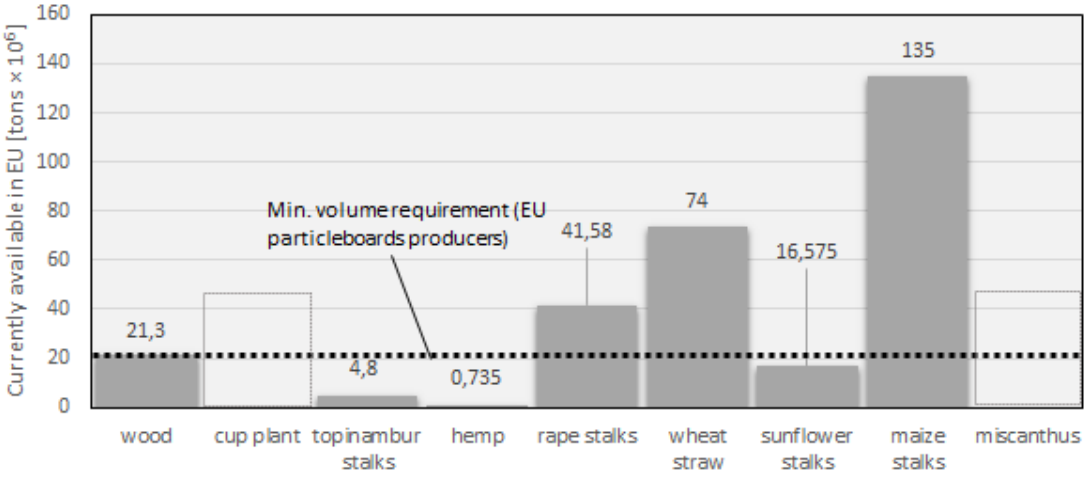

Fig. 2.
Alternative materials for particleboard production (calculated from data in Table 1, ha × t/ha). Empty columns represent cultivated energy crops.

Straw particleboards

One of the most important food plant that is feeding more than 40% of the world´s population is rice (Pathak et al. 2006; Mohdy et al. 2009). There are numerous examples for non-food utilization of the rice straw. A study on the production of ceiling boards using rice husks was presented by Ajiwe et al. (1998). Replacements wood by rice straw at 10% have shown increases in modulus of rupture (MOR) and modulus of elasticity (MOE). However, MOE and MOR dropped when the wood-replacement by rice straw was above 20% (Yang et al. 2003). Sound absorption panels using rice straw, produced from fine particles at 0.8 mm mesh size have turned out to be of higher performance than plywood (Yang et al., 2003). Internal bonding (IB) of rice straw particleboards has proportionally decreased as larger-sized straw particles were used, while MOR and MOE were proportionally higher. To fulfill EN 312 requirements, particles obtained from sieving with mesh sizes between 6.35 – 12 mm were found as ideal for the production of polymeric diphenylmethane diisocyanate (pMDI)-bonded rice straw particleboards. Urea formaldehyde (UF)-bonded rice straw particleboards, however, did not meet the requirements for IB and MOR, irrespective from the investigated particles geometry. Nevertheless, IB and MOR of rice straw UF particleboards can be increased by a pretreatment of rice straw using oxalic acid and steam, even though the average MOR was not high enough to meet EN 312 standards (Li et al., 2011). pMDI bonded rice straw particleboard mixed with coir fibers had higher IB and lower MOR and MOE (Zhang and Hu, 2014), compared to 100% rice straw particleboards. The particleboards from different parts of the rice straw particleboards without binder were suggested as well. The IB of these panels was way below the EN 312 requirements, however, results suggested that rice straw stalks are better suited for particleboards than rice leaf sheaths or leaf blades (Kurokochi and Sato, 2015). Industrially promising results are presented with low density insulation particleboards made from rice straw (density 220-360 kg/m^3), showing thermal conductivities between 0.046 – 0.060 W·m^{-1}·K^{-1} (Wei et al., 2015). These panels are competitive with low-density fiberboards made from wood.

Wheat straw was also used as a wood-replacement in particleboards. UF-bonded particleboards made from wheat straw particles met the requirement of class P1 in EN 312, with a pressing time of 320 seconds, and a UF resin content of 12 – 15 % (Boquillon et al., 2004). MOR, MOE and IB of wheat straw-based particleboards proportionally increased with higher UF resin content. MOR and MOE of UF-bonded wheat straw particleboards were further improved by mixing them with waste veneer splinters (Azizi et al., 2011). MDI-bonded wheat straw particleboard easily met the requirement of the EN 312. Properties can be substantially increased by bleaching wheat straw particles prior to the particleboard production (Mo et al., 2003). Aside of UF resin and pMDI resin, soybean protein resin was tried to produce wheat straw-based particleboards. Here, soybean resin-bonded particleboards did not meet the minimal requirement of P1 class in EN 312. Nevertheless, MOR, MOE and IB of soybean resin-bonded wheat straw particleboards reached similar values than those bonded with UF resin. Soybean flour-bonded wheat straw particleboard with a density of 840 kg/m^3 did meet the minimal requirements of P1 in EN 312, however, MOR was found to be weak (Cheng et al., 2004). Wheat straw was used to produce panels of lower density (Wang and Sun, 2002) for potential insulation purposes, using tannin-based adhesive (Tabarsa et al., 2011), or also a soybean protein-based adhesive (Cheng et al., 2004). Naturally PF-bonded particleboards from wheat straw fulfilled the minimal requirements by EN 312, even with 30 % of PF resin replaced by a tannin extract (Tabarsa et al., 2011).

In Poland, MDI bonded particleboards with straw coming from rapeseed (*Brassica napus* L.) stalks were suggested (Dziurka and Mirski, 2013). The particleboards from rapeseed straw were produced with densities between 350 - 550 kg/m^3. The 550 kg/m^3 pareseed panel fulfilled the requirement of P1 class in EN 312. It has to be noted that the pMDI dosage was 10 %, which was almost twice the commonly used dosage in industry. The rapeseed straw particles were also mixed with polystyrene particles in a ratio of 93 %^{wood}: 7 %^{polystyrene}, and bonded with MUF (Dziurka et al., 2015). Although these panels were produced at various densities (550 – 650 kg/m^3), the MOR of the panels was not satisfying with respect to EN 312, for boards with a density below 600 kg/m^3.

Plant stalks particleboards

Grigoriou and Ntalos (2001) investigated potentials of Castor stalks (*Ricinus communis L.)* for the production of particleboards. Findings have shown reduced values for IB, MOR, MOE and the screw withdrawal resistance, when castor particles replaced wood at amounts between 25 – 100 %. The conclusion was that wood can be replaced by 50% castor stalks and satisfying properties as defined in P1 of EN 312 can still be achieved. Cotton (*Gossypium hirsutum* L.) stalks-based particleboards (Guler and Ozen 2004; Khanjanzadeh et al. 2012) with density of 600 and 700 kg/m^3 also fulfilled the P1 class in EN 312. Cotton stalks-based particleboard properties proportionally declined as the density of the panels was gradually lowered.

Particleboard from sunflower (*Helianthus annuus* L.) stalks (Bektas, 2005) mixed with wood were also investigated. Mechanical properties of these boards were proportionally lower with the gradual substitutions of wood (Klimek et al. 2016). However, particleboards with 50 % sunflower particles replacing wood does meet the general purpose class in dry conditions (P1, EN312). Low IB was found to limit standard applications of the PF-bonded 100%-sunflower particleboards (Khristova et al., 1996). Particleboards from sunflower stalks with densities between 150 and 200 kg/m^3 were suggested for insulation purposes (Binici et al., 2014; Mati-Baouche et al., 2014). Thermal conductivity was 0.0058 W·m^{-1}·K^{-1}, which is lower than the one measured for conventional insulation wood-based fiberboards (Troppová et al. 2014).

Low density particleboards made with topinambour (*Helianthus tuberosus* L.), Miscanthus (*Miscanthus × giganteus*) and maize stalks were used in furniture production (Balducci et al., 2008; Dix et al., 2009). A wall-cabinet was produced from three layer particleboard, with Miscanthus or topinambour as the core material, and spruce as the surface layers (Dix et al., 2009). As a result, alternative shelfs-fastening was suggested for the cabinet, to compensate for the reduced IB and fastening withdrawal capacity. Some stalks from lesser known plants were also used. For instance, roselle stalks (*Hibiscus sabdariffa*) mixed with wood met requirements of P1 in EN 312 (Ghalehno and Nazerian, 2011; Klimek et al. 2016).

Extensive research on *Sorghum* stalks (Khazaeian et al. 2015) was reported with respect to the effects of particles sizes, press temperature and pressing time on mechanical properties. Three-layer tomato (*Solanum lycopersicum* L.) stalks (Guntekin et al. 2009) particleboards prepared with melamine-urea-formandlede (MUF) or UF adhesive have shown lower MOR, which restricted their applicability according to P1 (EN312). Particleboards from tobacco stalks (Acda and Cabangon 2013) were produced in several alternatives with tobacco stalks gradually replacing wood. Mechanical properties were decreased as wood was gradually replaced by tobacco stalks. However, termite resistance of these particleboards significantly improved: The fully wood-based panel type has shown a 100 % mass loss after 24-weeks of termite exposure, while tobacco-based (100 %) particleboards had almost no mass loss (~1.5 %). Buckwheat (*Fagopyrum esculentum*) stalks (Oh and Lee, 2012) were found suitable for particleboard manufacturing, if mixed with wood. Particleboards produced from buckwheat stalks only displayed lower MOR values, which restricted the range of applications. A similar behavior was found for particleboards produced from vine (*Vitis vitifera* L. CV. *sultani*) stalks/pruning (Ntalos and Grigoriou, 2002; Yeniocak et al., 2014). Also, UF-bonded particleboards from eggplant (*Solanum melongena* L.) stalks (Guntekin and Karakus, 2008) were not suitable for standard purposes due to low MOR and IB. Pepper (*Capsinum annuum*) stalks based particleboards did meet the required IB and MOE levels, however, MOR did not pass the minimum requirements (Guntekin et al., 2008). Lower mechanical performance was also found for particlebaords produced with cup-plant stalks (Silphium perfoliatum L.), with properties still at acceptable levels. Especially the pMDI-bonded particleboard types were found to be a viable alternative to classical UF-bonded particleboards made from spruce (Klimek et al. 2016).

An interesting approach for alternative particleboard was introduced by Selinger and Wimmer (2015). A novel sandwich hemp-based particleboard covered with pressed hemp-fibers nonwovens did meet the minimum requirement, although density was only 320 kg/m^3. The review by Youngquist et al. (1994) also mentioning possibilities of using cornstalks (*Zea mays* subsp. *mays* L.), mustard stalks, sugarcane (*Saccharum officinarum*), stalks cassava (*Manihot esculenta*) stalks, banana stalks, tapioca stalks or Ragweed (*Ambrosia* L.) stalks for particleboards.

Prunings particleboards

Various other parts of the trees, bushes, or waste materials were investigated to replace wood in particleboards. Kiwi (*Actinidia sinensis* Planch.) prunings (Nemli et al. 2003) replaced wood in particleboards and these panels had similar MOE and IB than wooden particleboards. Green vine pruning (Yeniocak et al. 2014) were used to gradually replace wood in particleboards. Interestingly, when vine prunings replaced the wood up to 70%, the mechanical performance remained unchanged. Nevertheless, particleboards prepared completely with vine prunings shown reduced mechanical properties, all below the minimal requirements of EN 312 - P1 class. A better performance was shown using tree prunings and branches. Particleboards made from prunings of native willow (*Acacia salicina*), buttonwood (*Conocarpus erectus*), council tree (*Ficus altissima*), white leadtree (*Leuceana glauca*), manila tamarind (*Pithecellobium dulce*), saltcedar (*Tamarix aphylla*) (Nasser 2012) were produced and their properties were similar to those made from spruce.

Bio-based wastes particleboards

Various by-products obtained from the food-industry were investigated to replace wood in particleboards. Gradual replacement by poppy husks (*Papaver somniferum*) proportionally decreased mechanical properties (Keskin et al. 2015). Particleboards with a poppy husks content above 25 % did not meet the requirements for class P1 in EN 312. Particleboards with rice husks (Ajiwe et al. 1998; Ciannamea et al. 2010; Suleiman et al. 2013) showed a low IB, which limited their applications according to P1 in EN 312. Rice husks were bonded with UF and phenol formaldehyde (PF) in amounts of 6%, 10% and 12 %, but IB did not pass P1 requirements, even when mixed with higher amounts of wood (75 $\%^{wood}$:25 $\%^{rice\ husks}$). When rice husks underwent an alkali treatment or bleaching, the properties substantially increased above P1 (Ciannamea et al. 2010). Çöpür et al. (2007) prepared hazelnut husk-based particleboards with densities of 600 kg/m^3 and 700 kg/m^3. Both types fulfilled the minimal requirements for particleboards used for general purposes (P1, EN 312). Particleboards were also produced from macadamia shell (Wechsler et al. 2013), bonded by castor oil based adhesive. Their properties did not fulfill P1. As a benefit, macadamia-shell particleboards displayed significantly lower thickness swelling than wood-based particleboards.

Particleboards made from almond and walnut shells displayed lower thickness swelling, reduced water absorption and also lower formaldehyde emissions, compared to the wood-made boards (Pirayesh and Khazaeian 2012; Pirayesh et al. 2013). The almond and walnut shell-made panels did meet requirements for particleboards used for interior fitments (P2, EN 312). In the Middle East, particleboards from date palm biomass have been produced. Palm's trunk and rachis particles were bonded with MUF and PF (Amirou et al. 2013), and the physical and mechanical properties of palm trunk-made particleboards were higher than those made with palms' rachis. At the same time, PF bonded rachis particleboards along with both types of palm trunk particleboards fulfilled P1 standrads, while MUF bonded rachis particleboard did not reach the P1 requirements.

Klimek et al. (2017) have successfully shown that particleboards may be also produced with 10% of brewer spent grain (BSG) at acceptable properties. BSG is a major by-product of the beer industry, representing around 85% of the entire by-products produced (Mussatto et al. 2006; Thiago et al. 2014). In the European Union BSG is generated at amounts of 3.4 million tons per year (Stojceska et al. 2008). While 10% substitution of wood by BSG particles did not change significantly the properties of the produced particleboards, higher contents of BSG particles resulted in reduced MOR, MOE, and IB, while thickness swelling and water absorption were increased. This fact is attributed to a different inner structure of the particleboards. It was found that smaller BSG particles tend to cover surfaces of wooden particles, while others fill voids between wooden particles. BSG-based particleboards have shown reduced performance, however, 10% of BSG did meet the requirements for general purpose particleboards used in dry conditions.

PET-flake particleboards

A wood- polyethylene terephthalate (PET) composite boards was produced, with up to 30% of PET flakes. IB, MOR and thickness swelling were assessed. It was found that the decline of mechanical properties can be mitigated by an air plasma pretreatment of the PET flakes. The samples with 30% of plasma treated PET exhibited the same internal bonding strength as the wood-based control panel. Panels with 15% of plasma treated PET exhibited the same modulus of rupture (Klimek et al. 2015).

Comparing particleboards made with alternative materials

The mechanical properties of particleboards produced from alternative resources were compared with respect to the related panel densities. The comparisons provided specific insights for particleboards made with alternative materials, which allows and easier visual comparison of properties. As seen in Figure 3 and 4, MOR of particleboards from agriculture residues do not display trends such as higher MORs with increasing densities. Strictly speaking, alternative particleboards produced at higher panels densities do not necessarily result in higher MOR. For instance, MOR of high-density (~900 kg/m^3) particleboards from oil palm leaves (Hashim et al. 2010 a,b) or particleboards from tissue paper mixed with corn peel (Lertsutthiwong et al. 2008) did not reach the P1 class in EN 312.

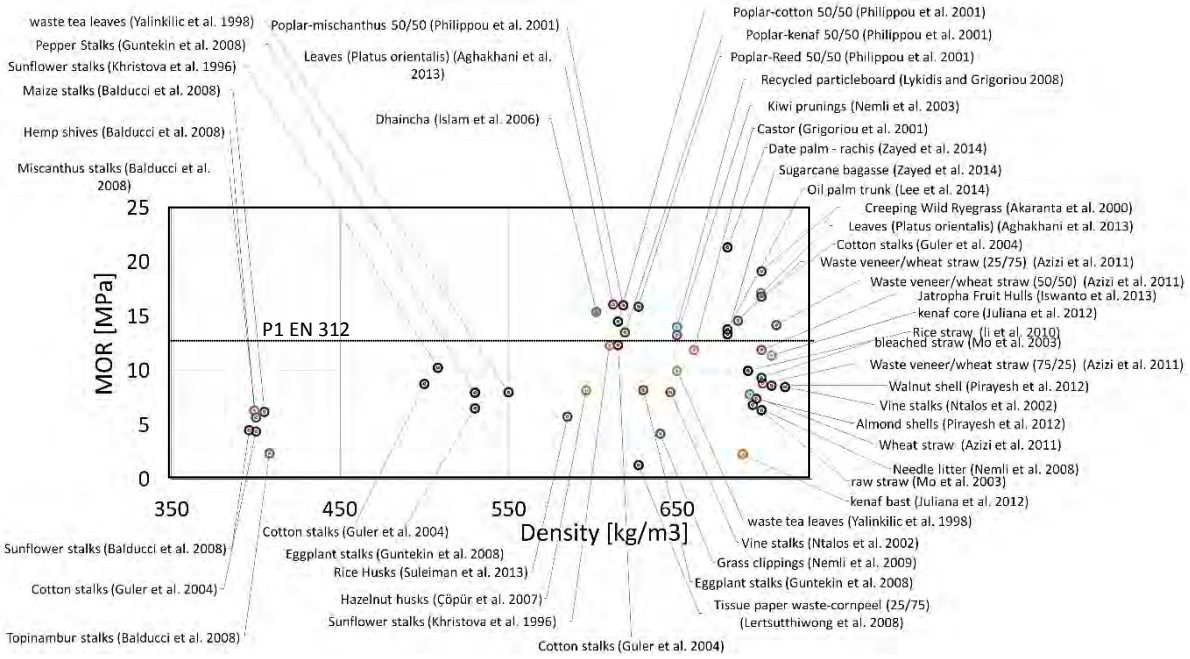

Fig. 3.
MOR – Panel density chart for particleboards made with alternative raw materials, for the panel density range 350-700kg/m^3.

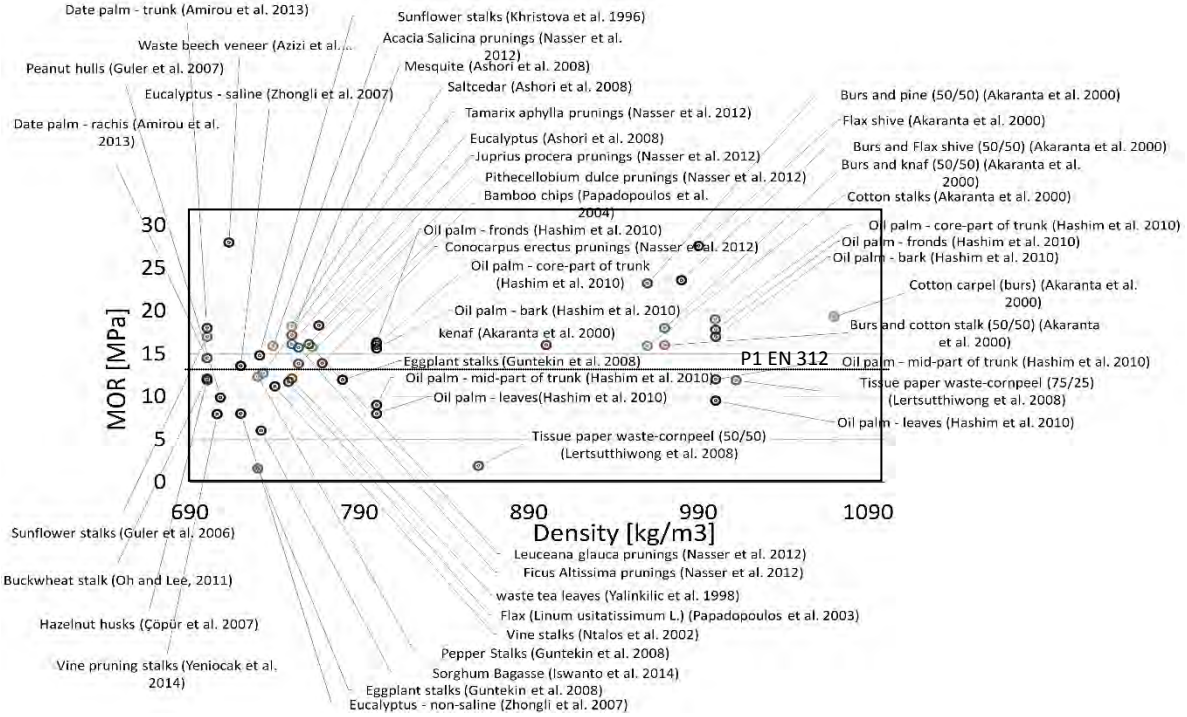

Fig. 4.
MOR – Panel density chart for particleboards made with alternative raw materials, for the panel density range 700-1090 kg/m^3.

MOE values (range 1000 – 3000 MPa) for particleboards made from alternative materials were plotted along with the related density of the produced panels in Figures 5 and 6.

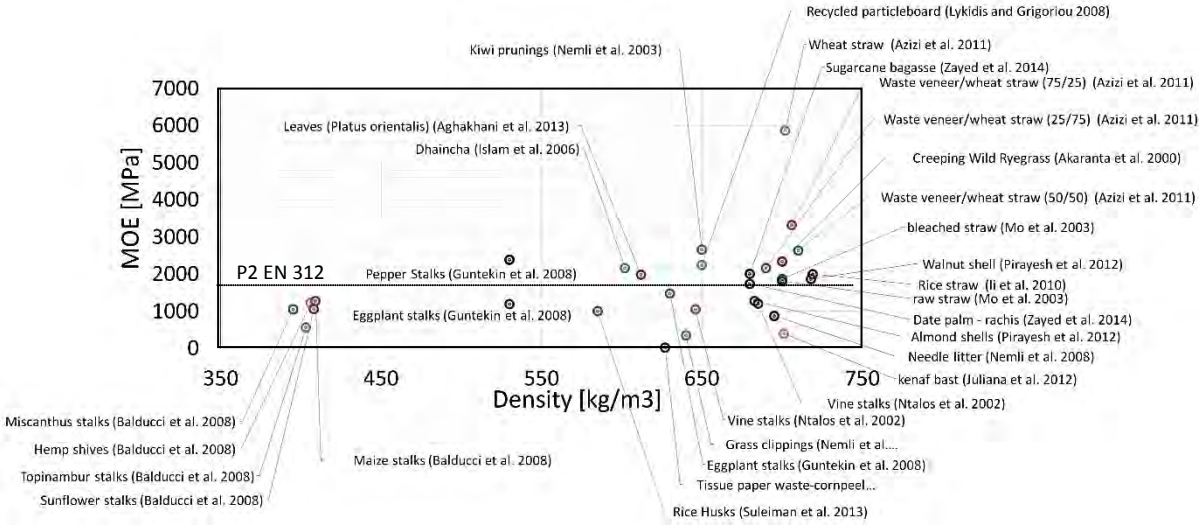

Fig. 5.
MOE – panel density chart for particleboards made with alternative raw materials, for the density range 350 - 700 kg/m^3.

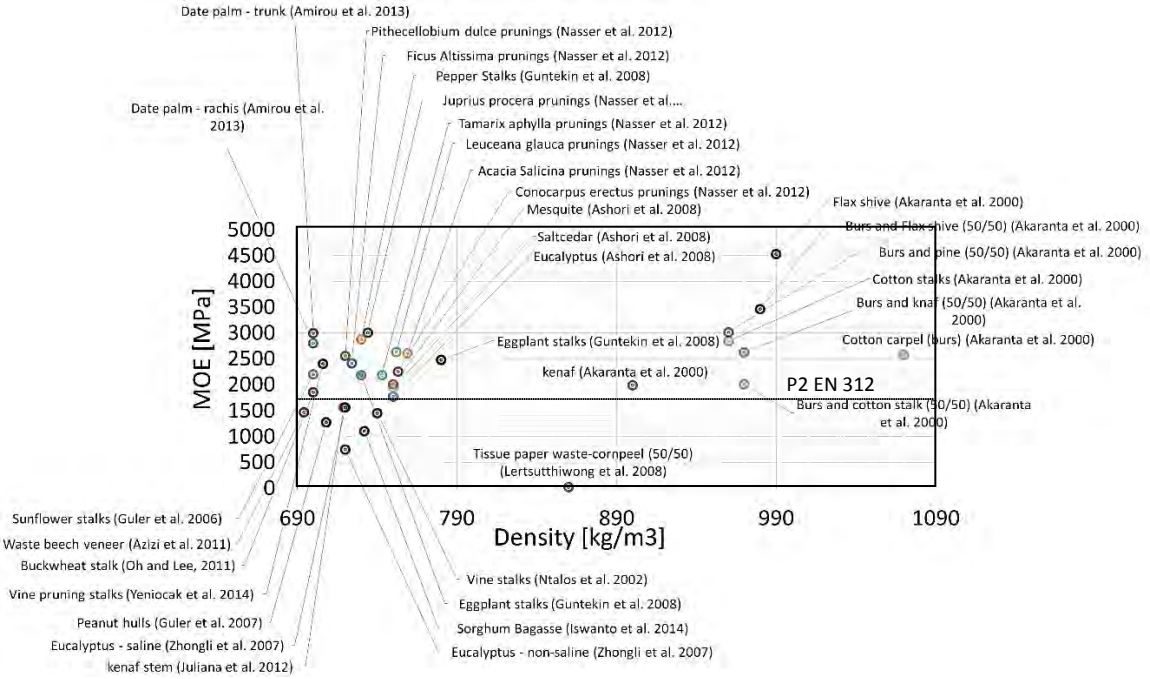

Fig. 6.
MOE – panel density chart for particleboards made with alternative raw materials, for the density range 700 - 1090 kg/m^3.

IB seems to increase slightly with particleboard densities. IB determined for the 550 – 700 kg/m^3 (Figure 7) range include about 50 % of the particleboards made with alternative resources, which ranke below the requirements of P1 in EN 312. For densities above 700 kg/m^3, about 15 % of the particleboards had insuficcient IB values (Figure 8).

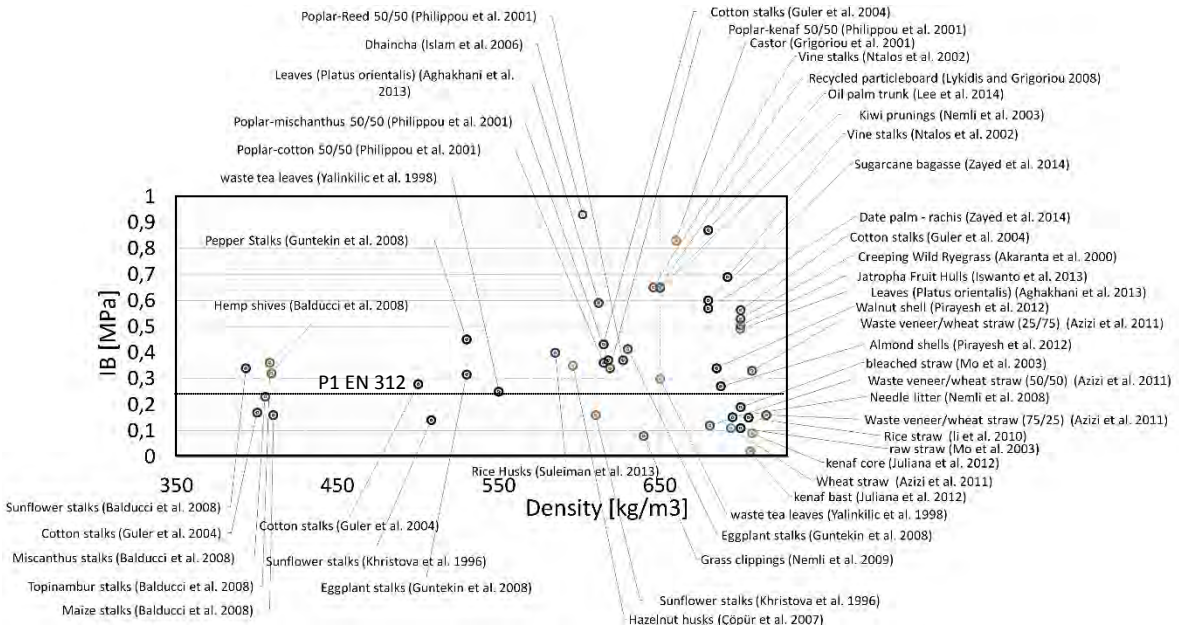

Fig. 7.
IB – panel density chart for particleboards made with alternative raw materials, for the panel density range 350 - 700 kg/m^3.

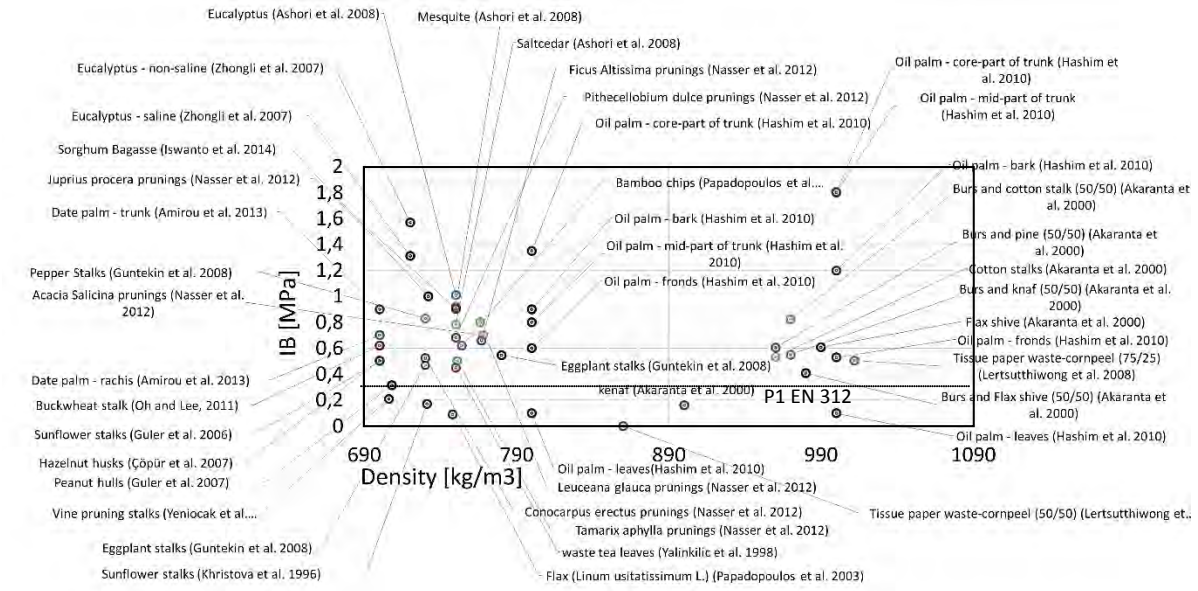

Fig. 8.
IB – panel density chart for particleboards made with alternative raw materials, for the panel density range 700 - 1090 kg/m^3.

Alternative material selection

The important point for material selection is to identify the suitability of the produced particleboards, and provide also information on possible constrains. EN 312 provides the requirements for standardized classifications of particleboards that producers usually follow. In this research, general purpose particleboards used in dry conditions (class P1) are defined.

Ashby ranking

To produce Ashby plots all compiled particleboard data were divided in four material classes: (1) Particleboards from plant´s stalks, (2) wood, (3) wood prunings, (4) straw and leaves. Data analyzed in following chapter were obtained from the reviewed literature. MOR in the Ashby graph (Figure 9) showed that groups of particleboards from wood, wood prunings, plant stalks and waste

leaves are overlapping. This means that these alternative particleboards could be produced with similar MOR like wooden ones. Concurrently, isolated groups of straw based particleboards and husks and hulls based particleboards indicated that MOR of these particleboard types need to be improved.

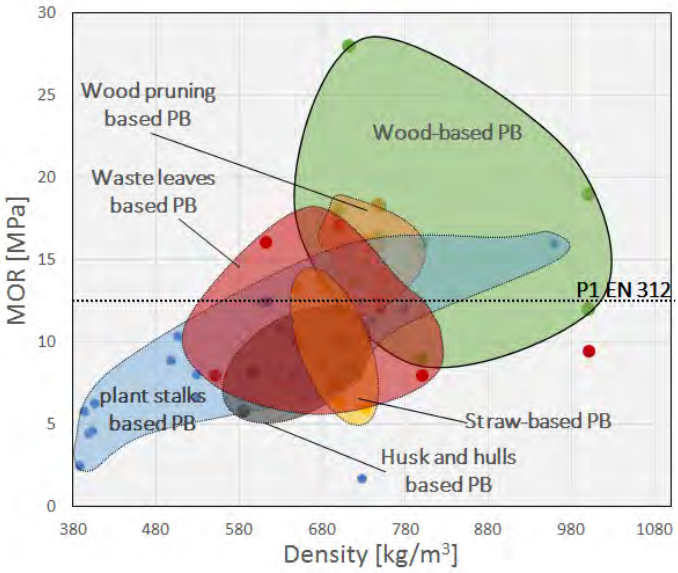

Fig. 9.
Ashby plot of alternative particleboard, showing MOR along with panel density.

MOE of stalks-based particleboards could be similar to wood-based ones, as these two groups are overlapping (Figure 10). The same is true for wood prunings-based particleboards. The isolated group refer to husks based particleboards, their MOE need to be improved with different actions (adhesives, sandwich constructions, mixing with other materials).

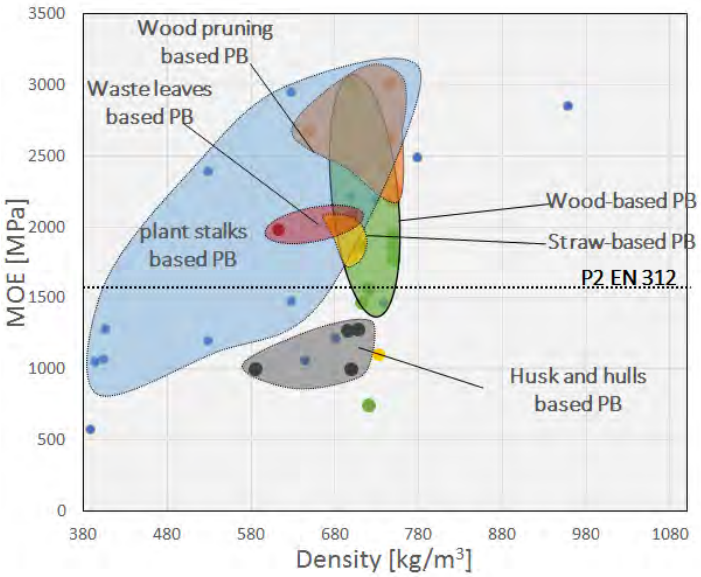

Fig.10.
Ashby plot of alternative particleboard´s MOE, and panel density.

Particleboards made from wood prunings and plant stalks may reach a similar IB than wood-based particleboards. IB of particleboards made from husks and hulls, waste tea leaves and straw needs to be improved. Nevertheless, the boards meet the requirements of the class P1 of EN 312. As shown in Figure 11, particleboards from wood prunings, stalks and waste leaves could be produced at the requirements of P1 class in EN 312, while particleboards from husks and hulls or straw are outside

the region meeting minimal required values. This is also illustrated with the IB-MOR graph (Figure 11 right)

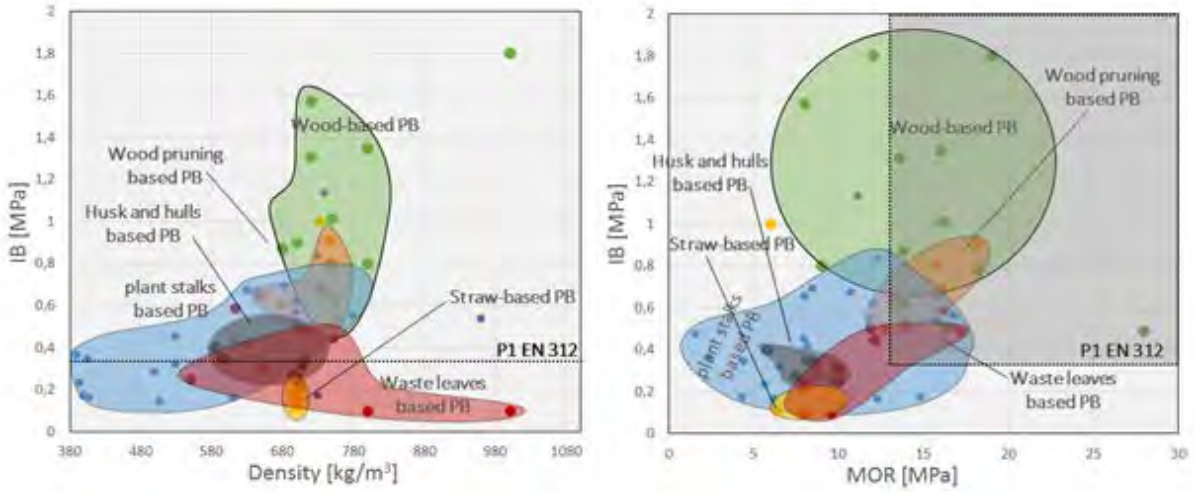

Fig. 11.
Ashby plot of alternative particleboard´s IB, plotted with panel density (left) and with MOR (right).

CONCLUSIONS

This literature review and assessment delivered the following conclusions: (1) Selected alternative materials are available at sufficient volumes and in some cases with even higher yields than wood. (2) Alternative materials may be significantly cheaper than wood. (3) Particleboards made from alternative materials may have additional benefits such as termite resistance, or lower swelling, however, mechanical properties are ususally lower compared to wood-based particleboards. (4) Particleboards made from plant stalks and prunings may have similar mechanical performance as wood-based ones. (5) Particleboards from alternative materials widely show satisfying mechanical performance at lower prices, compared to those made from wood. While wood has fully developed supply chains, alternative materials have supply chainds that are non-existient or only established rudimentarily. Strong logistics strutures, proper storage and sustainable raw material quality is highly needed and should be developed for alternative raw materials.

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Standards

EN 312 Particleboards and fiberboards – determination of swelling in thickness after immersion in water. 1993.

EN 310 Wood-based panels – determination of modulus of elasticity in bending and bending strength.

EN 319 Particleboards ande fiberboards – determination of tensile strength perpendicular to the plane of the board. 1993.

EN 312 Particleboards – Specifications.

TRENDS OF THE WOOD PRODUCTS HIGHER EDUCATION IN NORTH AMERICA

Marius Catalin BARBU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: cmbarbu@unitbv.ro

Timothy Mark YOUNG
The University of Tennessee, Center for Renewable Carbon
2506 Jacob Drive, Knoxville, Tennessee 37996, USA
Tel: 001 865946 1119, E-mail: tmyoung1@utk.edu

Salim Selim HIZIROGLU
Natural Resources Ecology and Management, Oklahoma State University
6013 Stillwater, Oklahoma 74078, USA
Tel: 001 405744 5445, E-mail: salim.hiziroglu@okstate.edu

Abstract

Wood products curriculum and education in North America have been changing last two decades. Some of the universities are loosing their wood products programs while some are merging with other bio-named departments. It is still fact that wood products graduates are still in demand but with a different perspective than several 10-15 years ago. It is important that expectation of industry should be taken into consideration to revise and develop new curriculum of the current programs. This article briefly reviews some of the wood science programs in North America and their background within the scope of overall trend and development.

Key words: *wood science; forest products and timber technology programs; higher education.*

INTRODUCTION

Forest products are commodities that have been used by mankind for thousands of years. Main forest products industry including sawn wood, wood based panels, pulp, and paper has an important market share of overall economy [Bowyer, 2000; Barbu and Paulitsch, 2016]. A learnt profession has something to do with a developing body of knowledge, formulation of ethical standards to conduct a certain performance, and to recognize a common interest using this knowledge in the form of service to the others in a society [Ellis, 1964]. In that respect forest products, specifically the wood products profession plays an important role in North America. It is a fact that the history of the forest products education and research in the form of wood science is less than a century old [Perlin, 1991]. Of course wood products education in North America has very significant role on overall utilization of forest resources within the scope of conservation and sustainability [Goodel, 2013].

Wood science is not a very versatile professional relative to other recently developed new professions such as food science, biosystems, and material science. However, there is quality research and public service from wood scientists which is a significant accomplishment given the many programs with many students and only a small contigent of faculty to serve the large audience of wood science programs. Many graduates from these programs play a significant role in leadership on the forest products industry. Currently there are about two dozen universities and two national laboratories in the USA and Canada in wood products education and research [Goodel et al., 2010, Barbu and Goodel, 2017].

OBJECTIVE

Objective of this article is to emphasize overall trends in wood science education with a brief review of programs at universties in the USA and Canada.

INSTITUTIONS FOR WOOD SCIENCE HIGHER EDUCATION IN USA AND CANADA

There are approximately ten institutions in the US having programs specialized in different aspects of wood science and forest products. The rest of the programs are mostly represented under different departments such as "natural resources" or forestry with only a few faculty members with limited number of courses and teaching forest products to forestry major students. Since, enrollment in forest products has dropped by over 60 to 70%, graduate programs are also reduced. As a result of

this trend some schools are under pressure to eliminate the forest products program. There are seven universities with only one wood products faculty member in the "natural resources" or forestry program. It is highly expected that these positions will be eliminated due to budget cuts in the next decade. These eliminations will play an important role in reshaping forest products education. More than 70% of forest products faculty positions have been elimnated in the US. Even though there is excess demand for forest products students by the industrial sector, many companies in the US have turned to hiring industrial engineering students to fill this gap. It is well known fact that long term basic research is very critical to forest product industry in the US. Financial support by government and private industry for forest product research has been declining last two and the half decades. Some of these forest product companies have eliminated research facilities. The demand for forest products is estimated to increase 25-30% by the year 2030 and in order to satisfy this there will be a definite need for education and research programs in forest products. Countries outside the US are supplying most of the graduate students in US forest products programs. Some of these programs are accredited by the Society of Wood Science Technology (SWST). Different actions need to be taken to reshape forest products programs in the US:

- Development of new products in the area of fiber enforced engineered materials with collaborations with other departments such as biosystems and other engineering disciplines.
- Rebuilding the relationships between wood products programs and industry to meet the actual needs of the industry. This should also fill the absence in theoretical research that is fundamental for applied research required by the industrial sector.
- Revamping curriculums in wood products program based on the needs of society and industry [Goodel et al., 2010; Goodel, 2013, Barbu and Goodel, 2017].

Some of the universities having wood science program in the US and Canada are briefly described described below:

Department of Wood Science and Engineering, College of Forestry, Oregon State University, Corvallis

Founded in 1868, Oregon State University has a student body of over 30,600 in a two campus system in Corvallis and Bend Oregon. Students can choose from more than 200 undergraduate and more than 80 graduate degree programs in 11 colleges, including over 20 degrees offered online.

The College of Forestry was established 1913 and currently the program has over 1,000 students, about 820 in undergraduate and 205 in graduate courses, offering 20 degree programs in the following four departments: Forest Engineering, Resources & Management, Forest Ecosystems & Society, Wood Science and Engineering, and the National Center for Advanced Wood Products & Design (NCAWPD). The NCAWPD was established 2015.

The Department of Wood Science and Engineering (WSE) is a very rare name in USA keeping "wood" in its name. Its mission includes educating new professionals for challenging careers, providing advanced learning for professionals in the work place, discovering new knowledge and solving problems through innovative research and transferring knowledge to users and practitioners. It is one of the largest and most diverse renewable materials programs in North America. WSE, well known for its industrial contacts, is offering a multi-disciplinary approach to the study of wood with increasing demand for new materials and intensifying global competition demand innovation, new discovery and well-educated professionals. The WSE research program areas are about the biodeterioration, materials protection, and product durability; composite materials; forest products business and marketing; green building and environmental performance -timber engineering, mechanics, and structural design but also wood aesthetics and natural coloration. The degree has changed to "Renewable Materials" for bachelor, and was kept as "Wood Science" for the master and PhD studies (about 35 students). Undergraduate enrollment is growing, currently at about 50. The faculty staff has 23 members, 15 of them with tenure, 7 adjunct or courtesy members and 3 office staff. Since 1999 about 60 graduates reached higher position in R&D at national and international level. In 1999, founded in Corvallis (OSU) and Blacksburg (Virginia Tech); the National Science Foundation (NSF) Industry/University Cooperative Research Center (IUCRC) for Wood-Based Composites having as partners the universities of British Columbia (UBC), North Carolina (NCSU), Maine (UM), Mississippi State (MSU), and other idnsutrial members. From 1927 to 2016, the WSE Department (and its predecessors) has granted the following degrees: 659 BSc, 388 MSc, and 106 PhD (numbers are approximate) [Barbu, 2017a, Oregon State Univ., 2016].

School of Forest Resources, College of Natural Sciences, Forestry, and Agriculture, University of Maine, Orono

The University of Maine was established as the Maine College of Agriculture and the Mechanic Arts under the provisions of the Morrill Act, approved by President Abraham Lincoln in 1862. In 1897 the original name changed to the University of Maine. The institution opened 1868 with 12 students and two faculty members. The Maine Agricultural and Forest Experiment Station was founded as a division of the University in 1887. In 1912 the Maine Cooperative Extension, which offers field educational programs for both adults and youths, was initiated. The first master's degree was conferred in 1881; the first doctor's degree in 1960. The University of Maine has an enrollment of 11,200 students in 2016.

In 1902 the Maine legislature granted money for "public education in forestry." It was used to start a Department of Forestry at the University of Maine. In 1993 the College was renamed and combined to form the College of Natural Resources, Forestry, and Agriculture.

The Wood Science option was consolidated into an undergraduate program Forest Operations, Bioproducts & Bioenergy (FBB) option in the School of Forestry in 2012. To meet society's increasing need for sustainable resources, efficient and environmentally acceptable options are needed for the management, harvesting, and transportation of timber and biomass for energy production and for manufacture of products, e.g. lumber, paper, and wood composites. The interdisciplinary BSc in Forest Operations, Bioproducts and Bioenergy (FBB) at the University of Maine aims to develop individuals (a) with the knowledge and abilities to better manage timber resources and forest operations in an environment of increasing public scrutiny and environmental concern; (b) with an understanding of the processes and challenges related to the efficient and environmentally acceptable harvest and conversion of forest resources to bioproducts and bioenergy; and (c) with an appreciation for the business principles and the associated local, regional, and global markets. The Forest Operations, Bioproducts & Bioenergy program is accredited by the Society of American Foresters and by the SWST. The graduate program in Wood Science and Technology at the University of Maine provides opportunities to earn either Master of Science or doctoral degrees. There are opportunities for research in wood to energy conversion, renewable nanomaterials, wood composites, and cellulose nanocomposites. There are several research centers at the University of Maine including the Advanced Structures and Composites Center and the Forest Bioproducts Research Institute. Graduates find work in industry, academia and government. Only five students at present enrolled at FBB for the Wood Science option [Barbu, 2017a, Univ. Maine, 2016].

Department of Sustainable Biomaterials, College of Natural Resources and Environment, Virginia Polytechnic Institute and State University, Blacksburg

Founded in 1872, Virginia Tech has the largest number of degree offerings in this state, more than 125 campus buildings, off-campus educational facilities in six regions, a study-abroad site in Switzerland, and agriculture research farm near the main campus, which is located in Blacksburg. Virginia Tech offers 215 undergraduate and graduate degree programs to more than 31,000 students (82.5 % undergraduate; 58.3 % male) and manages a research portfolio of more than $450 million. It is ranked 41st in university research in the United States.

Virginia Tech's College of Natural Resources and Environment (CNRE) is the only college specializing in natural resource education, research, and outreach in this state. The CNRE contains four different departments: Geography, Forest Resources and Environmental Conservation, Fish and Wildlife Conservation, and Sustainable Biomaterials. These departments currently offer eight undergraduate majors and a variety of options.

Established in 1979 as the Department of Wood Science and Forest Products (WSFP) in the College of Agriculture, the department has matured and grown and is now one of four departments in the CNRE. The first Department Head was Prof. Geza Ifju, who grew the department from 4 faculties in 1979 to 12 when he retired in 2000. In the 1980s through the 1990s, the WSFP department grew also in enrollments from a handful of BSc students to close to 100 students - the largest program in the country at that time. The following 10 years, enrollment of BSc students in the department steadily declined until 2010 (21 students for the 17 faculty members in the department). The department is recognized at the state, national and international levels as a leader in the education of highly-qualified professionals in the discipline. Renamed some years ago from WSFP in Department of Sustainable Biomaterials (DSB) and restructuring two new degrees it increased the enrollment by eight times the number of undergraduate students to approximately 170. The DSB is the largest department of enrolled students in the US. The program grewas soon as the new name was available and the two new education program with more "sustainable" and "greener" options were promoted. The addition of the green packaging degree that also emphasizes "design" which is very popular in the new name of

that bachelor degree "Packaging Systems and Design" has also helped. About 100 students are in the "Packaging Systems and Design", and 70 students in the "Sustainable Biomaterials" bachelor degree. 35-40 are graduate students at master and PhD level. The faculty of DBS is composed of a body of professionals counting 10 professors, 2 emeriti, 5 adjunct professors, 3 associate professors, 2 assistant professors, 3 senior researchers and 6 permanent staff in administration and technique [Barbu, 2017b, Virginia Tech, 2016].

Composites Materials and Engineering Center, Voiland College of Engineering and Architecture, Washington State University, Pullman

Washington State University (WSU) is a public land-grant university whichwas founded in 1890 and currently services around 25,000 students (52% women, 29% from abroad). With facilities throughout the State of Washington, the main campus is in Pullman (20,000 students). WSU has 11 colleges that foster scholarly achievement. Graduate and professional programs attract top minds from 79 countries. The R&D expenditures were over $300 million. The university offers 90 academic majors for undergraduates, 76 master's degree programs, 64 doctoral programs, 3 professional degree programs an over 20 online degree programs. 10 professors are National Academy members.

The Composite Materials and Engineering Center (CMEC) is comprised of an interdisciplinary facility administered through the Voiland College of Engineering and Architecture (VCEA) at WSU. For over 70 years the Wood Materials and Engineering Laboratory (WMEL) was established in 1946 exclusively for research in the Washington State Institute of Technology with Prof. George Marra as its 1st director. It became a part of the Department of Material Science and Engineering in 1972, and then in 1985, it was established as an independent laboratory within the College of Engineering and Architecture. WMEL discovered more efficient use of the forest and creative ways to transform wood waste into useful building products and developed areas of specialization in particleboard, fiberboard, waferboard, oriented strand board, and medium density fiberboard technology.

Pullman became a gathering place for the world community involved in wood and lingo-cellulosic composites, particularly at the annual International Washington State University Particleboard/Composite Materials Symposium (IPCMS) established 1967 by Prof. Thomas Maloney with about 500 people from 30 countries in each spring (today in Seattle known as "International Wood Composites Symposium" still the unique event of North America in this field but at less than the half no. of participants). The advanced degrees awarded up until this time were primarily master in Material Science and Engineering, and Doctorates in Engineering Science. The book of Maloney "Modern Particleboard and Dry-Process Fiberboard Manufacturing", which is also used throughout the world and continues to be requested and often referred today as the "Bible" in this sector of the forest products industry. WSU's educational programs for wood materials and engineering is unique in the United States with its administrative home in VCEA. Graduate students are generally admitted through the departments of Civil and Environmental Engineering, Mechanical and Materials Engineering, Chemical Engineering, or Biological Systems Engineering; as well as the interdisciplinary Materials Science and Engineering PhD. In 1996, the degree was changed through Civil and Environmental Engineering. More than 150 advanced degrees have been earned by students who worked under the guidance of the CMEC [Barbu, 2017a, Washington State Univ., 2016].

Wood Research Laboratory, Department of Forestry and Natural Resource, College of Agriculture, Purdue University, West Lafayette

Purdue University in West Lafayette (Indiana), was founded in 1869. Total enrollment in 2015 was 40,500 (30,000 undergraduates). Almost 30% of students are enrolled in the College of Engineering, but Purdue University's College of Agriculture (with 8% enrolment) is one of the world's leading colleges of agricultural, food, life, and natural resource sciences.

Forestry instruction at Purdue University began in 1905 with a two-course sequence titled "Forestry" which was taught in the Biology Department. In 1914, forestry courses were listed for the first time under the heading "Forestry" and was recognized as the beginning of forestry at Purdue University. An authorization was received from the Graduate School in 1944 to offer a Master's degree in forest production, forest economics, forest management, forest mensuration, silviculture, wildlife, and wood technology. The same year, the Wood Research Laboratory was established in the Forest Products Building. Authorization for offering a PhD program was received in 1961. Its current name The Department of Forestry and Natural Resources (FNR) was adopted in 1974. The FNR undergraduate enrollment peaked at 666 students in 1977 while graduate enrollment rose to about 80 students, as it is also today. Currently, the mission of FNR is to train the next generation of professionals in natural resource sciences and sustainable biomaterials process and product design.

The Wood Research Laboratory (WRL) is the unit of the FNR responsible for conducting research and teaching in wood science. Forest products research began at Purdue in 1904 when it became one of several timber testing stations under contract with the U.S. A Wood Technology curriculum was established in 1944. At this time, a dry kiln was constructed to research the drying of walnut blanks used for gun stocks and with experiments on special oak stock for making practice artillery shells. In 1960, Prof. Michael O. Hunt undertook extensive studies relating to the use of particleboard and related materials. Also Prof. Stan Suddarth received a grant from the US Forest Service for a goal to automate the kiln drying process. In 1962, Prof. Carl Eckelman undertook research related to the strength design of furniture. In general, the WRL had many pioneering contributions to wood science such as: wood truss design for residential and light-frame industrial buildings; evaluation of strength properties of structural lumber using machine stress grading; design and evaluation of wood-based composite products as well as development of application for their use; product engineering, quality improvement, strength design of furniture and its performance. Current research projects conducted at WRL are about log scanning, lumber processing, school furniture for developing countries, building construction from small diameter and low value timber, etc. Presently, the WRL is responsible for undergraduate and graduate wood science education including undergraduate majors in Sustainable Biomaterials - Process and Product Design; and two minors, Wood Products Manufacturing Technology and Furniture Design. The WRL is also responsible for graduate studies and offers MSc or PhD programs in Wood Science and Technology. Currently, it is a small program of about 10 to 15 undergraduate students in Sustainable Biomaterials - Process and Product Design Major and just about same number is enrolled in Furniture Design and Forest Products Manufacturing minors. In general, 5 to 10 graduate students are enrolled in the Wood Science and Technology. The WRL staff consists of 4 professional faculty members, 1 laboratory technician, and 1 clerical support [Barbu, 2017b, Purdue Univ., 2016].

College of Agriculture, Natural Resources and Design, School of Natural Resources, West Virginia University, Morgantown

West Virginia University was founded in 1867. The State of West Virginia was formed the following year and, shortly thereafter, the state's legislature accepted the terms for the Morrill Act to raise the money to start the new land-grant college they called the Agricultural College of West Virginia. In 1868, the school's name was changed to West Virginia University (WVU). Currently, WVU has 14 colleges and schools offering 353 majors. Hundreds of distance education and online classes are available. WVU enrollment is approximately 30,000.

The WVU Wood Science and Technology (WST) program is an option within the School of Natural Resources at the Davis College of Agriculture, Natural Resources and Design. The WST program was first accredited in 1989 and was re-accredited in 1999 when it was the bachelor degree in Wood Industries. The name of the program was changed to Wood Science and Technology in 2004 and reaccredited by SWST under that name in 2009. The WST Program also prepares students for careers in the production of wood products such as architectural woodwork, furniture, cabinetry, composite materials, and engineered wood products. The graduate WST program offers two levels of advanced degrees the Master of Science in Forestry (MScF) with an emphasis in WST, and the PhD. The overall WST educational program is currently stable at 25 undergrad students, 10 graduate students and 6 faculty members. The faculty are moving forward with a modified curriculum and a new program name in time for the next accreditation visit in 2019 [Barbu, 2017b, West Virginia Univ., 2016].

Center for Renewable Carbon, College of Agricultural Sciences and Natural Resources, University of Tennessee, Knoxville

The University of Tennessee, Knoxville (UTK), highest ranked public university in this state, was founded 1794 and is a public, comprehensive sun- and land-grant university. Today, the university offers education across nine undergraduate colleges and 11 graduate colleges. The main campus is located in Knoxville. The university counts among the nations elite research universities with more than 300 degree programs and spends more than $200 million annually on research. The university co-manages the Oak Ridge National Laboratory (ORNL) which is the US Department of Energy Science and energy laboratory with an annual budget of $1.4 billion. The university hosts 28,000 including 22,100 undergraduates. In the 2017 universities ranking, U.S. News & World Report UT ranked as 103rd among all national universities and 46th among public institutions of higher learning. In total, 87% of undergraduates are from the state of Tennessee, while 15% of graduate/professional students are international (over 90 countries).

The College of Agricultural Sciences and Natural Resources (CASNR) is a four-year institution with seven academic programs in a variety of natural, food, and social sciences; it also hosts the states School of Veterinarian Medicine. In the Department of Forestry Wildlife & Fisheries (FWF), part of CASNR, 241 undergraduates, 33 masters and 20 PhD students are enrolled. In particular, for the major in forestry, 79 undergraduates, 10 masters, and 14 PhD students are currently enrolled. FWF offers a single MSc and PhD major programs in natural resources. For undergraduate students, majors are offered in forestry resources management, urban forestry, and wildland recreation.
The Center for Renewable Carbon (CRC) is a research center positioned within the The Universiy of Tennessee, Institute of Agriculture (UTIA) of which CASNR administers the teaching responsibilities. The CRC conducts advanced interdisciplinary research in the field of biobased materials and provides innovative solutions to global challenges in energy, engineering by utilizing resources. The CRC has been involved in research in the past decade exceeding $300 million in funding levels. Research in advanced bio based materials includes raw material processing, characterization and product development in order to investigate economic viability of nanocellulosic materials, chemicals and fuels derived from renewable sources. The CRC had a unique program in the US in process analytics and statistical process control which has trained more than 1000 industry personnel in the last decade. The CRC has eight tenured faculty, two non-tenure research assistant professors and one distinguished professor. Since 2012, MSc and PhD academic degrees are awarded in forestry with a concentration in "Bio based Materials and Wood Science Technology" [Barbu, 2017b, Univ. Tennessee, 2016].

Department of Bioproducts and Biosystems Engineering, College of Food, Agricultural and Natural Resource Sciences, University of Minnesota, St.Paul

The University of Minnesota was founded as a preparatory school in 1851, seven years before the territory of Minnesota became a state. Over a century ago, when the University's Forestry Program began, Minnesota's natural resources looked vastly different than now. Mindful of these changes, the Department of Forest Resources has been continually evolving.

College of Food, Agriculture and Natural Resource Sciences (CFANS) created in 2006 via the merger of two colleges and a department consists of 12 academic departments and 10 research and outreach centers across Minnesota. The college offers degrees in 13 undergraduate and 13 graduate majors plus more than 25 minors. The Natural Resources Science and Management (NRSM) Graduate Program is among the top ranked programs of its kind. The National Research Council's latest rankings, released in 2010, place the NRSM program as high as number two nationally. Eight areas of study encompassing graduate course offerings from the Departments of Forest Resources; Bioproducts and Biosystems Engineering; Fisheries, Wildlife and Conservation Biology; and other units are offered. With 100+ students enrolled in the NRSM program, the student body represents a wide variety of educational backgrounds, geographic origins, and career objectives.

The Department of Forest Resources has 22 professorial faculties with 21 of these being tenured or tenure-track. FR also has 28 full-time research, scientist, and teaching staff that play a major role in carrying out the department's mission. Forest Products track is designed for graduate study specializing in areas such as: wood and fiber as raw materials; deterioration of wood; wood mechanics and structural design; wood moisture interactions and drying; processing and performance of composites; economics of manufacturing systems; technology and processing of solid wood products; marketing, design and production of housing components; and energy-efficient building construction [Barbu, 2017c, Univ Minnesota, 2016].

Department of Sustainable Bioproducts, College of Forest Resources, Mississippi State University, Starkville

The university began as the Agricultural and Mechanical College in 1862. The College received its first students in the fall of 1880. The School of Forest Resources had been established in 1954. The Department of Forestry began in the same year within the College of Agriculture. In 1955, the first two professional forestry degrees were awarded. In 1961, the School of Forestry became a separate entity from the College of Agriculture.

The Department of Sustainable Bioproducts began in 1964 as the Forest Products Utilization Laboratory as authorized by the Mississippi Legislature. In 1967, the Department of Wood Science and Technology was established and approved to offer master's degrees. The bachelor's degrees in wood science and technology was approved in 1975. The Furniture Research Unit was created in 1987 to support the burgeoning furniture industry in Mississippi, the number one producer of motion furniture. In 1989, the Department of Wood Science and Technology was changed to the Department of Forest Products. Most recently, in 2013, the department name changed to the Department of

Sustainable Bioproducts to reflect the renewable, natural and sustainable resources used in the industry. The Franklin Furniture Institute has tested furniture for every furniture manufacturer in the nation. Scientists in the department hold a record number of patents for termite control, wood preservation, non-destructive testing, and the development of new products. Graduate study in the Department of Sustainable Bioproducts leads to the MSc degree in forest products or a PhD in forest resources with an emphasis in forest products. Major areas of study include composite wood products, environmental biotechnology, wood preservation, business and production systems, wood chemistry, and furniture. Graduate research assistantships are available and include tuition waiver and medical insurance reimbursement for in and out of state students. The Forest Products degree was consolidated into an option within Forestry. In 2017, the department had 32 graduate students (9 MSc, 23 PhD) from 14 countries, and 10 undergraduates enrolled in the new program in biomaterials. The faculty is comprised of 6 professors, 2 associate professors, 5 assistant professors, one assistant research professor, 8 emeritus professors, and 7 adjunct faculty [Barbu, 2017a, Mississippi State Univ., 2016].

Department of Forest Biomaterials, College of Natural Resources, North Carolina State University, Raleigh

Founded in 1887, when the first class of 72 students enrolled, North Carolina State University (NCSU) counts nowadays more than 34,000 students and nearly 8,000 faculty and staff. NC State's research expenditures are approaching more than $325 million annually, with almost 70% of faculty engaged in sponsored research and 2,500 graduate students supported by research grants. NCSU is ranked third among all public universities in industry-sponsored research expenditures. It is ranked among the US top 50 public universities and ranked by Princeton Review as a best value for students. The departments, Forestry and Environmental Resources, Parks, Recreation and Tourism Management, and Forest Biomaterials are ranked among the nation's best in their fields.

The Department of Forest Biomaterials (FB) is part of NCSU's College of Natural Resources (CNR). It was founded in 1929 as the NC State's School of Forest Resources and is one of the oldest and largest departments of its kind in the nation. In 1948, the Division of Forestry established its first wood utilization program, featuring a curriculum centered on wood technology and lumber products merchandising. A year later, formal research programs and laboratory teaching methods were introduced in order to provide students with a way to combine their fundamental knowledge with practical skills better preparing them to join the ranks of industry upon graduation and to enjoy success in their careers. By 1950, MSc degrees and PhD's in Wood Technology were available. In 1951, a special pulp and paper program that combined the School of Forestry, Department of Chemical Engineering and Department of Chemistry was announced. Meanwhile, a wood products laboratory had been built and equipped through funding and donations. More mill working equipment and furniture manufacturing machines were donated, and a second wood products laboratory was created. Other facilities for both wood science and paper science areas followed in rapid order, including the Hodges Wood Working Laboratory and the Robertson Laboratory of Pulp and Paper Technology. In 1960, a fifth year program was inaugurated to allow students to obtain a BSc degree in pulp and paper technology in four years, complete the requirements for a BSc in chemistry engineering during a fifth year (a program that continues today). By the end of the 1960's, NC State had obtained the largest collection of tropical wood in the U.S. and become a leader in study, research and instruction involving tropical woods and their uses. The 1980's saw an increasing emphasis on environmental science and biotechnology in the wood, paper and pulp industries. Demand for NC State graduates exploded, and foundation support and endowed scholarships continued to grow, with 50% of all paper and pulp students receiving scholarships. By the mid-1980's (and continuing today), NC State had the largest undergraduate wood, paper and pulp student enrollment in the US. In 1999, as the wood products program continued to expand and support North Carolina's wood-related industrie, the paper science faculty significantly revised its undergraduate curriculum. In 2000, a grant was approved for a 100% distance learning-based MSc of Wood and Paper Science, the first pulp and paper degree of its kind in the world. In recognition of these research initiatives, the Department of Wood and Paper Science was renamed the Department of Forest Biomaterials in 2010. And in 2013 the Wood Products degree was expanded to explicitly include class in sustainability, and renamed to Sustainable Materials and Technology. Enrollment in a BSc in Wood Products Business Management and BSc in Wood Products Manufacturing & Engineering is currently at 36. FB is currently home to 20 full-time faculty, 12 active adjunct faculty, 9 staff members, 5 research associates, more than 150 undergraduate students and 48 graduate students, participating in both on-campus and distance education curriculum. It has over 2,000 alumni [Barbu and Goodel, 2017, North Carolina State Univ., 2016].

Department of Agricultural and Biological Engineering, College of Agricultural Sciences, Pennsylvania State University, Mont Alto

In 1855 the Commonwealth chartered the Pennsylvania State University (PSU) at the request of the Pennsylvania State Agricultural Society. Founded in 1930, the Department of Agricultural and Biological Engineering in Penn State's Colleges of Agricultural Sciences and Engineering, provides high quality engineering education and research. The educational programs offered are the BSc in Biological Engineering, BSc in Agricultural Systems Management, and MSc and PhD in Agricultural and Biological Engineering. Faculty have been consolidated into an Agricultural & Biological Engineering Department.

Wood Products will phase out as a new undergrad (and grad) program in Biorenewable Systems is developed that will retain a "wood" core, but also embrace other bioproducts. Currently Wood Products has about 20 students [Barbu, 2017c, Pennsylvania State Univ., 2016].

Departments of Forest and Natural Resources Management and Paper and Bioprocess Engineering, State University of New York, Syracuse

The State University of New York (SUNY) was founded at Potsdam, New York in 1816. Since 1948 SUNY has grown to include 64 individual colleges and universities that were either formerly independent institutions or directly founded by the State University of New York. SUNY provides access to almost every field of academic or professional study within the system via over 7,000 degree and certificate programs. Total enrollment is over 467,000. Nearly 40% of New York State high school graduates choose SUNY. The alumni number over 2.7 million graduates.

Department renamed to Sustainable Construction Management and Engineering (SCME) with the elimination of the Wood Products Engineering degree. The SCME are now offered through the departments of Forest and Natural Resources Management and Paper and Bioprocess Engineering. SCME has been dissolved as a department. It will continue to offer courses in topics of Wood Products Engineering for students interested in wood science, properties of wood as a construction material, wood identification, and materials marketing. The new department offers a BSc in Construction Management, and graduate degrees in Sustainable Construction Management and Wood Science. Graduate programs leading to MSc or PhD degrees are offered in three options: Construction Management, Sustainable Construction, and Wood Science. A concentration in Wood Products Engineering provides optional coursework in the manufacturing, properties and marketing of wood products. Currently SUNY has a Construction Management major. Enrollment has been stable at about 85 students [Barbu, 2017c, State Univ. of New York, 2016].

Department of Forest, Rangeland and Fire Sciences, University of Idaho, Moscow

The University of Idaho opened its doors in 1892, when it welcomed about 40 students and one professor. In 1896, the university graduated its first class when four students marched across a stage to receive their diplomas. Two years later, the university awarded its first graduate degree. Today, the university is home to nearly 12,000 students and nearly 3,159 faculty and staff. It continues to be a leading place of learning in Idaho and the West, because although it is ever-responsive to the changing needs of its students and society, it never forgets its roots and traditions.

The Department of Forest, Rangeland, and Fire Sciences gives a wide range of ecology and management skills pertaining to forests and landscapes. Graduates of Forest Resources degree program are extensively recruited by employers in public and private sectors. New degree in "Renewable Materials" in a Forest, Rangeland and Fire Dept. Already they are growing enrollment at 17 [Barbu, 2017c, Univ. of Idaho, 2016].

School of Forestry and Wildlife Science, Auburn University, Alabama

Auburn University today is a comprehensive grant institution helping fulfill the dreams of nearly 25,000 students. The university began, as the small, East Alabama Male College, which was chartered in 1856 and opened its doors in 1859 as a private liberal arts institution.

The School of Forestry and Wildlife Science offers degree program (MSc and PhD) in Forestry, Wildlife Sciences and Natural Resources, and a Masters of Natural Resources (MNR). Three MNR options are available: Natural Resource Management, Advanced Forestry Studies, Professional Forester Applied Economics [Barbu and Goodel, 2017, Auburn Univ., 2016].

College of Engineering, Forestry and Natural Sciences, Northern Arizona University, Flagstaff

Founded in 1899, Northern Arizona University (NAU) is a public university with academic programs, research, public service, and creative endeavors that enrich lives and create opportunities

in Arizona and beyond. NAU counts 151 number of degree programs, 31 average class size and 30,000 students enrolled.

At the College of Engineering, Forestry and Natural Sciences (CEFNS) the next-generation engineers and scientists are trained for careers. Degree programs are available for Forestry, International Forestry and Conservation, Forest Health and Ecological Restoration, Forest Science [Barbu, 2017d, Northern Arizona Univ., 2016].

Department of Natural Resource Ecology and Management, School of Forestry, Oklahoma State University, Stillwater

The Department of Natural Resource Ecology and Management (NREM) have expertise in conducting interdisciplinary instruction, research, and extension education which focus on the natural resources of fisheries, forests, rangeland, and wildlife within and beyond the boundaries of Oklahoma. The NREM faculty support undergraduate and graduate programs in Fire Ecology, Fisheries, Forestry, Natural History, Rangeland, Wildlife and Wildlife Biology and Preveterinary Science. The NREM curriculum prepares students to plan, implement, and research the management, protection, and sustainable use of natural resources. The department provides an integrated education in renewable natural resource management, conservation, and utilization, as well as a valuable perspective for understanding and solving critical contemporary environmental problems at local, regional, and global scales. NREM has 27 faculty members in different areas including one professor in wood science [Barbu, 2017d, Oklahoma State Univ., 2016].

College of Agriculture, School of Renewable Natural Resources, Louisiana State University, Baton Rouge

In the State of Louisiana, the forest industry contributes over 50% of the total value of all agricultural, animal and fish/wildlife commodities. In addition to lumber, plywood, OSB and the production of other primary products, valuable secondary products are also produced such as furniture and kitchen cabinets.

In 1994 the LSU Agricultural Center established the Louisiana Forest Products Laboratory (LFPL). In 2003 the name was changed to the Louisiana Forest Products Development Center (LFPDC) to better reflect the breadth of our expertise and client base. The Center, now an integral part of the School of Renewable Natural Resources, provides technical assistance to the primary and value-added processing wood products industries in Louisiana. Since its inception, the LFPDC has made great strides and is currently firmly positioned as one of the most recognized and productive forest products research and outreach centers in the United States [Barbu, 2017d, Louisiana State Univ., 2016].

Forest Products Laboratory, U.S. Department of Agriculture, Madison

Nation's only federally funded wood utilization research laboratory the Forest Products Laboratory (FPL) is primarily or partly responsible for many of today's wood-based technologies, including wood preservatives, glulam beams, oriented strand boards, and fiber-based packaging.

At the turn of the 19th century, logging had proceeded across much of the eastern United States and demands for wood products were rising rapidly. In 1910, FPL was established in Madison (Wisconsin), to find ways to conserve scarce timber resources. For almost 100 years, the mission of FPL has been to use the Nation's wood resources wisely and efficiently, while at the same time keeping our forests healthy. The research began with preserving railroad ties, and now we are venturing into nanotechnology and finding ways that our research can contribute to mitigating the impacts of climate change. Early research highlights of FPL were: the reduction of timber demand for railroad ties by 75% through preservatives research, the increase of average lumber yield per log from 25% to 60%, the use of wood frame technology used in over 90% of the Nation's homes and the first prefabricated home designed and constructed in US. The FPL research staff has the experience and expertise needed to make us world renowned among forest products research organizations and an unbiased source of information. FPL researchers, currently employed over 60 scientists with an average of 20 years of experience in their related fields. The range of wood research spans from fiber and chemical science to composites. Whether it's putting a self-adhesive, environmentally friendly stamp on an envelope or walking on a hardwood floor, FPL has in some way contributed to making those products and innovations [Barbu, 2017d, Forest Products Lab., 2016].

Department of the Wood and Forest Sciences, Faculty of Forestry, Geography and Geomatics, Laval University, Quebec City

Laval University, with 45,000 students enrolled at undergraduate and graduate levels in 2014, is the oldest francophone university in North America, founded in 1663. Faculty of Forestry, Geography and Geomatics (FFGG), founded originally in 1910 as School of Surveying and Forest Engineering has a total of 1,266 students enrolled in 2014, of which 959 are enrolled at undergraduate level in three departments (Geography, Wood and Forest Sciences and Geomatics), while 175 are enrolled at master and 132 at doctoral level. These students are educated by 67 professors of the FFGG, of which 31 are the professors teaching the students of the Department of the Wood and Forest Sciences. The FFGG has also 310 employees offering administrative and technical services, of which 181 are with DWFS.

The Department of Wood and Forest Sciences (DWFS) is the result of merger of three departments in 1985, those of Ecology and Pedology, Sylviculture and Forest Management and Exploitation and Utilisation of Wood, existing as such since 1965. The Wood Engineering Program (WEP) represents a unique engineering program in Canada, educating wood engineers who become members of the Order of Engineers of Quebec. This program is unique in Quebec and in Canada because it is the only WEP accredited (since 2002) by the Canadian Engineering Accreditation Board (CEAB) in Canada. The WEP (120 credits of which 90 in required courses and 30 in optional courses) is conceived as a program educating wood engineers through development of 12 engineer qualities within required (compulsory) courses, while the students are also offered a possibility of choice of 12 credits among optional courses in order to get access to 4 concentrations (mentioned in their diplomas if they choose that option) in: 1) Wood Construction, 2) Engineered Wood Products, 3) Green Chemistry and Biorefinery and 4) Industrial Engineering and Manufacturing Systems. WEP offered at Laval University is a cooperative program, which means that the students are required to perform 3 internships in industry (during fall, winter and summer trimesters) during their four years' curriculum, with a possibility to add yet a fourth (optional) internship. In 2014 a total of 48 students enrolled in WEP (undergraduate level) was recorded, while at graduate studies in wood sciences, 9 students were enrolled at master and 24 at doctoral level. These students are educated in an exceptional environment provided by the Research Center for Renewable Materials by 8 professors teaching in the WEP. The CRMR, founded 2013 in continuation of Wood Research Center founded in 2002, regroups 47 researcher regular members and 13 associated members from Laval University, University of Quebec in Trois-Rivières, Abitibi-Témiscamingue and Chicoutimi, as well as from two technical colleges, from FPInnovations, CECO bois and from Natural Ressources of Quebec and of Canada. There are a total of 179 graduate students and postdoctoral fellows associated to the CRMR [Barbu, 2016b, Laval Univ., 2015].

Department of Wood Science, Faculty for Forestry, University of British Columbia, Vancouver

The University of British Columbia (UBC) is a global centre for research and teaching, consistently ranked among the 40 best universities in the world. Since 1915 when UBC officially established with three Faculties: Arts and Science, Applied Science, and Agriculture, its West Coast spirit has embraced innovation and challenged the status quo. Its entrepreneurial perspective encourages students, staff and faculty to challenge convention, lead discovery and explore new ways of learning. At UBC Vancouver campus 25 faculties and schools are established. Today, UBC has over 61,000 students of which more than 13,000 are international coming from 155 countries and over 15,000 faculty and staff. The annual budget of the university exceeds the CDN $ 2.3 billion and it is ranked 34th in the world according to Times Higher Education for 2015.

Between 1915 to 1918 was officially established the Faculy for Agriculture which later hosted the first forestry course. In 1920, Department of Forestry was authorized at UBC and 1923 first BSc degree in Forest Engineering was awarded. Ten years later the first MSc degree in Forest Engineering was awarded. In 1949 the MSc of Forestry and PhD programs were authorized and in 1951 the Faculty of Forestry was established. 1957 signifies the welcoming of the Sopron students and staff to the Faculty of Forestry at UBC. The students of the Sopron School of Forestry in Hungary were forced to flee their homeland when the anti-Soviet Revolution failed. This mass immigration provided BC with a new perspective on forestry and a major contribution to the industry. In 1981, the Departments of Forest Resources Management, Harvesting and Wood Science, and Forest Science are established. One year later a major in Wood Science and Industry was introduced to BSc. In 1997, the BSc in Wood Products Processing was established. After 1990 a significant wave of graduates students from Eastern Europe join MSc and PhD courses of this faculty within UBC. In 2016, approximately 1,027 undergraduate students and 260 graduate students (about 50/50 split between MSc and PhD) are

enrolled at the faculty. Forestry has 61 professors and a number of instructors/sessionals that are responsible to deliver its six undergraduate programs.

The Department of Wood Science (DWS) was formed in 1982 as one of the three departments including Forest and Conservation Sciences and Forest Resources Management that comprise the Faculty of Forestry at UBC. The DWS offers both undergraduate and graduate programs leading to BSc, MSc, and PhD degrees, respectively. The undergraduate program Wood Products Processing (WPP) includes advanced wood products processing, industrial processing technology, business management and marketing, and science and engineering. Currently, the WPP has 170 students that also offers the option of co-op where students will spend periods of time with companies gaining practical experience and being paid for a total of at least one year. The graduate degrees are offered in many fields related to wood science & technology, biotechnology and the forest products industry, processing and business. All of the nineteen professors in the DWS supervise over 70 students in a 60/40 (PhD/MSc) split working towards their post-graduate degrees in wood science. The Centre for Advanced Wood Processing (CAWP) was created, in consultation with the University's Forestry Advisory Council, with input from the National Education Initiative on the Canadian Wood Processing Industry (NEI), to address the need for advanced technical and managerial training for the value-added wood products manufacturing sector. CAWP is an interdisciplinary initiative administered by the DWS. The CAWP supports part of the WPP program and provides industry support through consulting and R&D projects both at national and international level. Industry education is also a strong activity of this centre [Barbu, 2016b, Univ. of British Columbia, 2015].

Center of Integrated Wood Design, University of Northern British Columbia, Prince George

Located in northern British Columbia, UNBC is one of Canada's best small and research-intensive universities. UNBC provides undergraduate and graduate learning opportunities that explore cultures, health, economies, and the friendly, inclusive, and supportive environment. It was in 1990 that the members of the BC legislative assembly passed the UNBC Act, officially creating it. The dream of having a northern university goes back much further, to the 1960s, when the land on which UNBC's Prince George campus is currently situated as a university reserve.

The Master of Engineering Program is rooted in the specific needs to the British Columbian and North American wood construction industry, adding value from the sawmills to the building site, and well aligned with strategies and priorities that lead into sustainable construction. This program focuses on educating students in the field of modern wood structures including up-to-date structural design, seismic design and mixed structures, as well as building physics, energy efficiency and sustainability. The knowledge obtained in this program enables students to actively contribute to the evolution and innovation in the construction industry. The aim of the MSc of Engineering in Integrated Wood Design program is to educate and train engineers and qualified professionals. Students come from a variety of undergraduate programs in BC, Canada, and worldwide. The curriculum consists of 16 courses with a total of 51 credits over 12 months, developing integrated design and research skills. The entire program is offered in a block structure to increase efficiency and attractiveness for students, faculty, and international experts [Barbu, 2016b, Univ. of Northern British Columbia, 2015].

Forest Product Innovations, Vancouver

FPInnovations (FPI) is a not-for-profit world leader forestry research organization, which specializes in the creation of scientific solutions in support of the Canadian forest sector's global competitiveness and responds to the priority needs of its industry members and government partners. FPI was established in 2007 through an amalgamation of Forintek (established in 1918 as Forest Products Laboratories of Canada, which later became Forintek Canada Corp.), Paprican (the Pulp and Paper Research Institute of Canada, since 1925), Feric (Forest Engineering Research Institute of Canada, since 1975) and a partnership with CWFC (Canadian Wood and Fibre Centre of Natural Resources Canada). FPI is strategically positioned to perform research, innovate, and deliver state-of-the-art solutions for every area of the forest sector value chain, including forest operations and fibre assessment, primary and secondary wood products manufacturing, supply chain analytics and decision support systems, transport logistics and roads, advanced building systems, bio-based chemicals and biomaterials, biorefinery and energy, pulp and paper, packaging, consumer products and nonwovens.

FPInnovations has more than 500 skilled staff, working in its world-class R&D laboratories, pilot plants, and technology transfer offices across Canada in Montréal, Vancouver, Québec City, Thunder Bay, Hinton and Ottawa. FPInnovation is offering complete value chain solutions with an annual operating budget of about CDN $90 million, more than 400 industrial and government members across Canada, partnerships with more thn 25 universities universities and other institutions

in Canada and abroad. It has 300+ patents in key forest processing markets around the world, 100+ patented technologies and processes, 100+ R&D projects, seven pilot plants (durability and protection, composites, medium density fiberboard, wood engineering, lumber manufacturing, cellulose nanocrystals, lignin), about a dozen R&D programs, one pilot paper machine, and Canada's largest CT imaging centre. FPI has been a partner of many forest sector university networks such as ForValueNet, Value Chain Optimization, NEWBuilds, SENTINEL Bioactive Paper, Green Fibre, Biomass Conversion, Lignowork, ArboraNano, Fibre Centre, hosted at universities incuding Univ. Laval, UBC, Montreal, Toronto, Queens, Dalhousie, Lakehead, McMaster, Politechnique, McGill, UNB, UNBC, Alberta, UQAT, UQAM, among others [Barbu, 2016b, Forest Product Innovations, 2015].

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SECTION 1. WOOD STRUCTURE AND PROPERTIES

CHARACTERISATION OF ODORANTS IN WOOD AND RELATED PRODUCTS: STRATEGIES, METHODOLOGIES, AND ACHIEVEMENTS

Linda SCHREINER
Friedrich-Alexander-Universität Erlangen-Nürnberg, Professorship for Aroma Research, Emil Fischer Center, Department of Chemistry and Pharmacy, Henkestraße 9, 91054 Erlangen, Germany, Email: linda.schreiner@fau.de
and
Fraunhofer Institute for Process Engineering and Packaging IVV, Giggenhauser Straße 35, 85354 Freising, Germany, Email: linda.schreiner@ivv.fraunhofer.de

Jonathan BEAUCHAMP
Fraunhofer Institute for Process Engineering and Packaging IVV, Giggenhauser Straße 35, 85354 Freising, Germany, Email: jonathan.beauchamp@ivv.fraunhofer.de

Andrea BUETTNER
Friedrich-Alexander-Universität Erlangen-Nürnberg, Professorship for Aroma Research, Emil Fischer Center, Department of Chemistry and Pharmacy, Henkestraße 9, 91054 Erlangen, Germany, Email: andrea.buettner@fau.de
and
Fraunhofer Institute for Process Engineering and Packaging IVV, Giggenhauser Straße 35, 85354 Freising, Germany, Email: andrea.buettner@ivv.fraunhofer.de

Abstract

Wood is a ubiquitous material that has been used by humans for building construction, furniture, products of daily use, or in the form of derived products such as paper and cardboard, for generations. Extensive studies have been made on the emissions of volatile organic compounds (VOCs) from diverse types of wood, yet only limited information is available on their related odour-active constituents. This article reviews the current state of knowledge on wood odours, covering studies targeting odorants from different woods and investigations on the physiological impact of wood odours on human beings. Further, modern strategies towards elucidating odorants in wood and wood-derived products using analytical tools in combination with human sensory evaluations are introduced and discussed. Specifically, these methods are based on well-established odorant analytical methodologies that are routinely used in the field of food science, namely gas chromatography-olfactometry (GC-O) in combination with sensory dilution approaches such as aroma extract dilution analysis (AEDA). The implementation of such methods allows the relative odour potency of individual odorants within a sample to be ascertained and their identities to be determined via gas chromatography mass spectrometry/olfactometry (GC-MS/O) and two-dimensional GC-MS/O (2D-GC-MS/O). The successful identification of potent odorant constituents of wood offers insights into the molecular basis of wood odour profiles, which can be used to understand the transition of possible off-odours or woody smells in wood-related products.

Key words: *gas chromatography-olfactometry; mass spectrometry; odour-active; physiology.*

INTRODUCTION

Olfaction is one of the oldest human senses from an evolutionary perspective (Albrecht&Wiesmann 2006). The action of smelling proceeds when scent molecules that are present in inhaled air are transported to the receptor sites at the nasal olfactory cleft. Individual odorant molecules then interact with the array of >350 different olfactory receptors at the epithelium to trigger neurological signals that elicit an odour impression in the brain (Buck 2004). Olfactory perception exhibits a high level of complexity that results from manifold activation and inhibition patterns. On the one hand, certain odorants can act on more than one receptor, yet conversely there are specific receptors that become activated by more than one type of odorous molecules. To add to this, specific odorants can undergo metabolism within the nose prior to reaching the receptors, thereby increasing the complexity of interactions (Schilling 2017). When an odour-active molecule binds to an olfactory receptor, the intra-cellular enzyme adenylyl cyclase becomes activated in a process that is mediated via G-proteins. This enzyme catalyses the transformation of adenosine triphosphate (ATP) into cyclic adenosine monophosphate (cAMP), which acts as a secondary messenger and leads to the opening

of ion channels. Consequently, the resulting change in the cellular potential of the olfactory cell triggers a signal transmission to the brain, which is relayed in a complex pattern to different regions of the brain (Menini et al. 2004).

Human olfaction is generally underestimated, yet our sense of smell has many functions (McGann 2017). At the individual level, olfaction serves in subconscious chemo-communication between humans, whereas at the cellular level it facilitates signalling between individual cells within the human body. Notably, olfaction is an implicit aspect of our interaction with food, not only in providing a hedonistic parameter for food enjoyment, but also in relation to the detection and avoidance of spoilt food (Solov'yov et al. 2012). Besides food, humans regularly interact with many products that exhibit odour; wood is a raw material that can be included in this category. Humans are exposed to wood and related items on a daily basis, including furniture and construction materials, or derived products such as pencils, paper and packaging goods, to name but a few.

The odour of wood is typically described as pleasant with positive associations, and many consumers would preferably choose wood-based articles over those made out of synthetic materials. Despite its general positive appreciation, however, the exact nature of wood odour is largely unresolved, to date. The present study aimed to address this knowledge gap by reviewing wood odour-related studies and exploring the odorous constituents of wood and wood-based products by applying state-of-the-art odorant analytical tools. Specifically, several different wood species and diverse wood-based products such as cellulose fibres, pencils or paper and cardboard materials were investigated with respect to their odour and its composition. New insights arising from these studies can aid in understanding the formation pathways of individual odorous constituents in wood and help in implementing avoidance strategies of potential off-odour development in wood-based products. Furthermore, these data add to the scientific repository of the constituent odorants of wood and wood-based products.

Wood extractives – important fractions of wood constituents

Wood primarily comprises of the biopolymers cellulose, hemicelluloses, and lignin, with minor amounts of inorganic compounds and extractives. The latter consist of terpenoids, steroids, fats and waxes, and phenolic constituents (Sjöström, 1981). They contribute significantly to the odour of wood by being precursors of the odour-active compounds, thus the specific class of extractive under investigation is of high relevance when studying wood odorants. One study on extractives from the heartwood of five different Scots pine (*Pinus sylvestris* L.) trees, which were processed via Soxhlet extraction and subsequently analysed using gas chromatography-mass spectrometry (GC-MS), led to the detection of primarily fatty acids and resin acids (Ekeberg et al. 2006). In another study on this substance class, proteins, amino acids, fatty acids, terpenes, resin acids, steroids, and phenols were detected (Rowell, 2005). By comparison, the main lipid classes identified in lipophilic extractives from pine wood by GC-MS were fatty acids, resin acids, triglycerides, sitosterol, waxes, and sterol esters (Gutiérrez et al. 1998).

Wood essential oils

Wood essential oils, which are derived from extractives via steam distillation or hydro-distillation, are rich in wood odorants and have been the subject of several studies in relation to their odour composition. The essential oil of cedar wood (*Calocedrus decurrens (*Torr.) Florin) heartwood, for example, was found to be mainly composed of p-cymene and p-menthane derivatives (Veluthoor et al. 2011), sesquiterpene hydrocarbons, oxygenated derivatives and monoterpenes (Dai et al. 2013). Other studies focussing on the composition of essential oils from Pinaceae woods indicated that monoterpenes (hydrocarbons and oxygenated), sesquiterpenes, and diterpenes were the major constituents, thereby offering first insights into the relevance of terpenoid compounds to the wood odour (Garcia Vallejo et al. 1994; Radulescu et al. 2011; Salem et al. 2015).

Wood volatiles

Wood extractives are rich in volatiles, typically with several hundred different constituent VOCs in individual species. Although many of these are not odorous in nature, a broad overview of the volatiles hitherto detected in diverse woods offers insights into prospective main odour-active candidates that contribute to wood odour.

GC-MS analyses on wood extractives (distilled wood chips) of black pine wood and juniper species (Uçar&Balaban, 2002) revealed a total of 140 VOCs in juniper (Uçar&Balaban 2002) and over 200 in black pine tree (Uçar&Balaban 2001), most of which were monoterpenes, sesquiterpenes or other terpenoids. Other studies on Scots pine (*Pinus silvestris* L.) confirmed monoterpenes as being

the major group of wood volatiles (Flodin&Andersson 1977), but have also revealed the presence of many aliphatic aldehydes and alcohols (Weissbecker et al. 2004).

Volatiles of oak and comparable woods have been investigated extensively due to their common and widespread use in cooperage for the maturation of alcoholic beverages (Cadahía et al. 2003; Fernández de Simón et al. 2009; Vichi et al. 2007). Most related studies have focused on the volatiles in naturally-seasoned and toasted wood, which represent the common treatment conditions of woods used in barrel-making. As a result many of the reported substances are formed by degradation of wood lignin, carbohydrates and lipids and Maillard processes of the original wood constituents during these heat treatments. Compounds included a range of phenolic compounds, furan derivatives, lactones, alkyl aldehydes and ketones.

Despite the wealth of information from studies on volatiles in wood, in most cases these do not allow for the odorous character of the wood to be predicted.

Physiological effects of wood odour

The physiological influence of wood odour on animals and humans has been the focus of several investigations. In one study, the essential oil from Sakhalin fir (*Abies sachalinensis* (Mast.)) was observed to have an anxiolytic effect on mice (Satou et al. 2011). In another study, α-pinene, an important wood odorant, was observed to have an alleviating effect by suppressing stress-induced hypothermia in rats (Akutsu et al. 2002).

Such relaxing effects have similarly been demonstrated in other trials. The essential oil of Siberian fir (*Abies sibirica (*Ledeb.)), for example, was observed to reduce the arousal level in the panellists when performing visual exercises, as indicated by electrocardiogram and electroencephalogram signals of the panellists (Matsubara et al., 2011). In another study, an experiment that assessed subjective differences in feelings of wellbeing in a room equipped with walls of Japanese cedar wood compared to a control conditioned room observed that liking and feeling responses of the panellists were significantly different between the two conditions (Matsubara&Kawai 2014).

Different feelings of comfort have also been reported between forest and city environments. In one particular study on the physiological influences of such environments it was reported that cerebral activity and salivary cortisol levels reflected a relaxing influence for panellists in a forest environment compared to that of a city (Park et al. 2007). This effect might relate to the observation that pine needles have a potent antioxidant activity and can increase cell viabilities (Ka et al. 2005).

Investigations on wood odour

As indicated above, despite a series of focussed studies on wood extractives and volatiles, and physiological effects of wood odours, few reported investigations on the odour-active compounds in wood exist. Indeed, the odour of native wood has hitherto not been fully characterised. One particular study on this topic focussed on the most odour-active substances in various wood extracts, in particular in woods used for barrels in the aging of wine and spirits (Culleré et al. 2013). In that study, wood chips of acacia, chestnut, cherry, ash or oak were toasted with medium intensity and the samples were then analysed by GC-O and multi-dimensional GC. Different odorants were detected, including various phenolic compounds, as well as compounds arising from the degradation of wood carbohydrates and lipids. Due to the toasting process, however, it is unclear whether or not the respective odorous compounds result from the fresh and native wood or were formed during thermal degradation. Similar studies using GC-O and GC-MS were performed on toasted and non-toasted American, French and Russian oak woods (Díaz-Maroto et al. 2008). Many short- and medium-chain alkyl aldehydes, acids and alcohols, as well as various compounds with fruity and floral notes like linalool oxide, phenylethanol and trans-cinnamaldehyde, were found to be present in the toasted and non-toasted oak wood samples.

Overall, woods used to make barrels for the aging and maturation of alcoholic beverages have been the subject of studies on their odour-active constituents in view of exploring how these might affect the final beverage product. By comparison, odorant investigations on other types of wood, especially those used for furniture or other products of daily use, or in the paper and cardboard industry, are very scarce. Knowledge on the odorous constituents of wood, however, can be essential when trying to ascertain the source of off-odours in wood-based products and in the development of associated avoidance strategies. Such wood-based products include pencils, fibre materials, or packaging materials. Cardboard packaging has been the subject of a targeted odorant investigation in which a sensory analysis revealed that the most potent odours were cardboard-like, woody, and musty (Czerny&Buettner 2009). Aroma extract dilution analysis (AEDA) (Grosch 2001) and two-dimensional

GC-MS/O measurements indicated that these odours were caused by the presence of aldehydes and phenolic compounds (Czerny&Buettner 2009).

Release kinetics of volatile wood constituents

In terms of health and wellbeing, not only is the identification of the odour-active constituents of wood important, or an understanding of how these affect human physiology, but given their ubiquitous nature, it is also essential to ascertain their concentrations in different environments, especially in the indoor setting. Wood is a strong source of emissions in the indoor environment and a large contributor to overall indoor air quality (Weschler 2009), although most of the 'negatively' associated emissions from woods are from composite woods such as plywood, particleboard, fibreboard and oriented strand board (OSB), which utilise adhesive resins that can emit a mixture of aldehydes and terpenoids (Hodgson et al. 2002). In addition to classical VOC analysis using GC-MS, the use of on-line mass spectrometric approaches might in future offer insights into the release kinetics of individual odorants from wood and wood-based products. One such method is the chemical ionisation-based technique of proton-transfer-reaction mass spectrometry (PTR-MS), which is a quantitative, real-time analytical tool for characterising rapid concentration changes of VOCs (Hansel et al., 1995). The technique has been successfully implemented to investigate emissions from living trees (Williams et al., 2001), but studies on the kinetics of wood emissions are very limited. In one such investigation, emissions from OSB were comparatively studied using two types of PTR-MS instrument, namely a PTR-quadrupole-MS (PTR-QMS) and a PTR-time-of-flight-MS (PTR-TOFMS) (Schripp et al. 2014). The focus of those studies, however, was less on identifying individual compounds or characterising their emission kinetics, but rather to generate mass spectral fingerprints for use in quality control assessments. In another related study, the kinetics of the generation of secondary products of terpene oxidation – the latter being the primary compound class emitted from wood – under the influence of coexisting carbonyl compounds were investigated using PTR-MS (Ishizuka et al. 2010).

Despite the aforementioned kinetics-related investigations, there are hitherto no studies using on-line analytical tools that solely target the emissions of wood odorant constituents in view of their implications for daily exposure or wellbeing.

Identification of wood and related products – a new approach

Whereas the general framework on odorous wood constituents, namely wood volatiles, essential oils, and physiological effects, have already been the subject of several studies, only limited information is available on the odour-active substances. In an attempt to close this current gap in knowledge on wood odorants, we carried out comprehensive targeted investigations to sensorially characterise and identify the main odorants of wood and wood-based products in a range of non-barrel woods like incense cedar (Schreiner et al. 2017). We applied modern-odorant analytical tools, as routinely applied for aroma analysis of food, to investigate several types of wood as well as cellulose fibres and products made from wood.

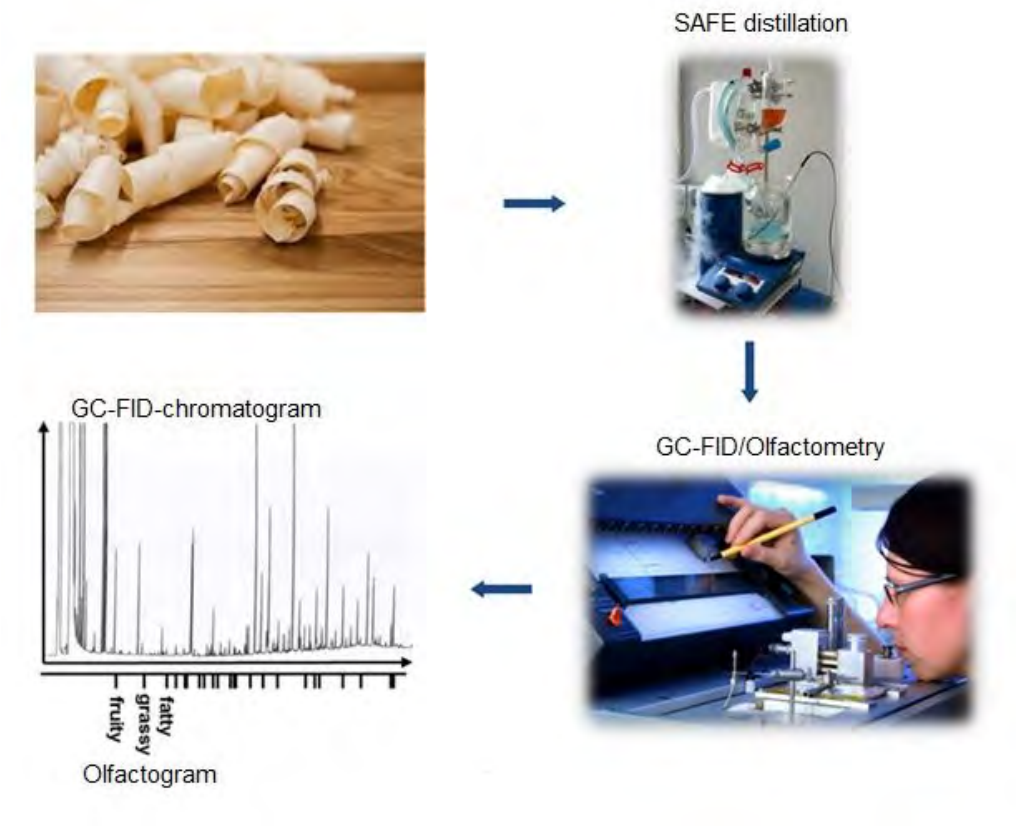

Fig. 1.
Typical steps in analysing wood odorants: solvent extraction and gentle distillation of the volatile fraction by solvent assisted flavour evaporation (SAFE) followed by gas chromatography-olfactometry (GC-O) (with flame ionisation detection; FID) to screen for the odorous compounds. The output of this analytical process is a chromatogram from the GC-FID, with signals relating to individual compounds eluting from the chromatographic column at different retention times, and an olfactogram, describing the odour impressions of individually eluting compounds, as perceived by the trained panellist at the odorant detection port. (© Fraunhofer IVV).

Figure 1 shows the typical steps undertaken during the targeted analysis of wood odorants. Volatile constituents of individual wood samples are extracted by solvent assisted flavour evaporation (SAFE) (Engel et al. 1999) and this extract is then further enriched by means of Vigreux distillation and micro-distillation (Bemelmans 1979). The gentle conditions for distillation (SAFE and distillation columns held at 55°C) reduce the likelihood that the odorant substances are lost in the extraction process (e.g., through degradation or evaporation) and avoid the potential generation of new odorants during distillation. The odorous substances are then analysed by GC-O (cf. figure 2) and are ranked according to aroma extract dilution analysis (AEDA) (Grosch, 1994). Compound identification is then made by GC-MS and two-dimensional GC-MS (2D-GC-MS/O).

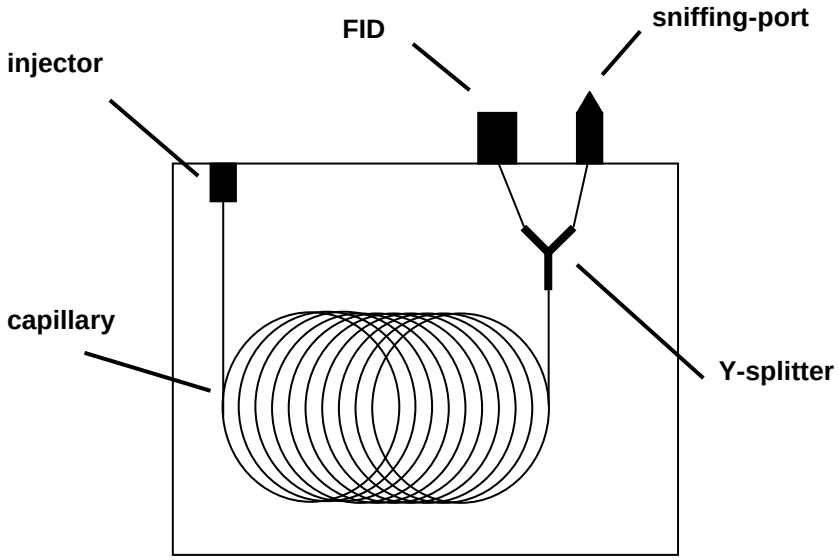

Fig. 2.
Schematic illustration of a gas chromatography-olfactory (GC-O) system with flame ionisation detection (FID) and simultaneous olfactory assessment via a sniffing port. (© Fraunhofer IVV).

After screening for odorants by GC-O, a more comprehensive analysis by 2D-GC-MS/O is carried out for purposes of compound identification and to target substances present at low concentrations or those that co-elute with other compounds (cf. figure 3). In the latter, the volatiles are pre-separated on a GC column and the odorants are detected via GC-O. Then, the specific odour compounds are transferred onto a second capillary of the 2D-GC-MS/O system. After separation, the odorants will be transferred after splitting the gas-stream between a detector (MS) and a sniffing port. The use of two chromatographs with analytical capillaries of different polarity leads to an increased separation efficiency and sensitivity.

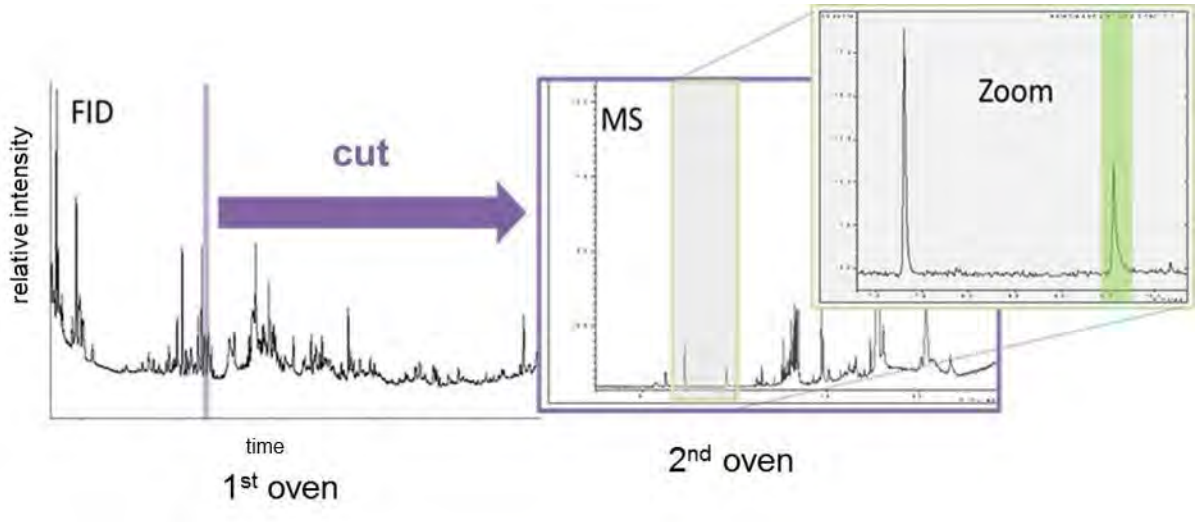

Fig. 3.
Separation and detection of odorants via two-dimensional gas chromatography-mass spectrometry/olfactometry (2D-GC-MS/O). A specific elution period from the first capillary (chromatograph oven) is cut and transferred to a second capillary of different polarity. (© Fraunhofer IVV).

If odorants remain unresolved after 2D-GC-MS/O analysis, structure-odour-relationships can be explored. This involves the synthesis of chemically-related substances of the known odorants and a subsequent investigation of their properties. Such structural effects on odour impressions have been comprehensively explored for fatty acid-derived compounds and terpenoids, as well as for aroma compounds that may contribute to the odour of wood and wood-based products (Elsharif&Buettner

2017; Lorber&Buettner,2015; Lorber et al.,2016; Schranz et al.,2017). Once the key parameters such as retention indices and odour quality are known, a prediction of an odorant's structure can be made, allowing for the synthesis of various related compounds and a directed investigation of the structure-odour relationships with a view to elucidating the identity of the target molecule.

In one study, we analysed the odour-active constituents of cedar wood, which revealed numerous odorous substances that represent various smells such as fatty notes, cheesy and fruity smells, as well as typical wood-like, green notes. Five compounds were detected for the first time as wood odorants in incense cedar, including the pencil-like smelling thymoquinone (Schreiner et al. 2017).

CONCLUSIONS

Wood is a natural product that is rich in VOCs, many of which are odorous, yet comprehensive knowledge on these odorants is limited, to date. We successfully applied combined human-sensory and odorant analytical methods to investigate the chemical structures of the main odorants in selected woods and wood-based products. After characterising the odour of the wood via sensory evaluations, samples are analysed by GC-O and are ranked according to odour potency via AEDA. The samples are then analysed using GC-MS and 2D-GC-MS/O to reveal the identities of the main constituent odorants. Although this analytical procedure is well-established in the field of food aroma analysis, it represents a new approach for elucidating wood odour, and indeed the use of SAFE distillation for wood products for a targeted odorant extraction is a new application in this field of research. The results of these studies can help to establish a wood substance library comprising data on substances identified in wood and wood-based products, their odour qualities, and their analytical properties such as linear retention indices and characteristic mass spectra. Such a library helps to create a better understanding of compounds that generate typical wood smells, and is an essential aid when assessing the potential daily impact of wood odorants on humans and their wellbeing.

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MECHANICAL CHARACTERISATION OF ACCORDIONISATED WOOD, EFFECT OF RELAXATION CONDITIONS

Mátyás BÁDER
University of Sopron, Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts
Str. Bajcsy-Zsilinszky nr. 4, 9400 Sopron, Hungary
Tel: 0036 99 518 187, Fax: 0036 99 518 647, E-mail: bader.matyas@phd.uni-sopron.hu

Róbert NÉMETH
University of Sopron, Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts
Str. Bajcsy-Zsilinszky nr. 4, 9400 Sopron, Hungary
E-mail: nemeth.robert@uni-sopron.hu

Abstract

After longitudinal compression the natural wood will be bent easier. The relaxation after compression results in better properties. The maximum deflection increases with the relaxation time and with the decreasing of the modulus of elasticity (MoE), while the needed force for the same bending also decreases. The maximum bending strength (MoR) changes intensively just with a long-time relaxation. The nanoindentation (NI) showed, that the changes can be found also inside the cell walls. The S2 cell wall layer's hardness slightly decreases, while their modulus of elasticity decreasing is remarkable.

Key words: *wood modification; longitudinal compression; accordionisation; mechanical properties; wood bending.*

INTRODUCTION

The longitudinally compressed wood can be bent with lower forces in smaller curves till the break. A more suggestive naming is "accordionisation", because the cell walls deform, crinkle during the process and finally seems like an accordion on the microscopic pictures (Fig. 1). The required bending force and the *MoE* decrease dramatically, and ensure a high deformability (Ivánovics 2006).

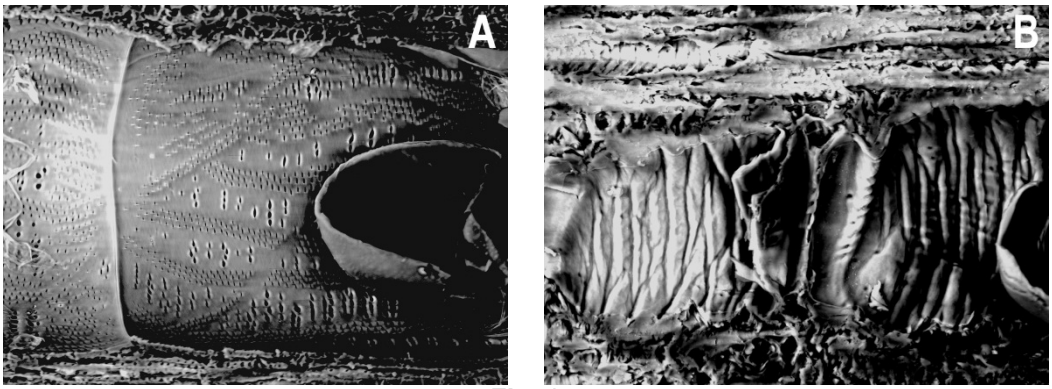

Fig. 1.
SEM photographs of the oak wood's trachea in case of 2 samples (20 kV x200): A – control; B – longitudinally compressed.

Mostly high quality hardwood raw material can be used for the process (Buchter 1993). Before the procedure the wood has to be plasticized by steaming. The raw material's moisture content should be at least about the fiber saturation point (Báder and Németh 2016).

To provide the desired mechanical properties in the sample, the compression rate has to be equal along the entire length. Having markings along the sample's length and measuring their distances before and after compression, the sections compression rate can be exactly specified.

In this experiment the samples compression rate was set to 20% according to their original lengths. Compression rate affect the modified wood's mechanical properties (Kuzsella 2011). After compression the sample can be hold for a while under pressure, this period is called relaxation. Mechanical test was carried out to get the mechanical influence caused by different relaxation times.

OBJECTIVES

This series of researches was made to evaluate the effect of the longitudinally compression and the effect of the relaxation time on oak wood and beech wood sample's properties. Untreated, steamed and accordionisated samples were compared with 1 minute, 3 minutes, on occasion 5 minutes and almost a day long relaxed wood samples. Such scientific examinations had never been published. This article is an overview of the preliminary results.

MATERIALS AND METHODS

Accordionisation

In this experiment Sessile Oak (*Quercus petraea* (Matt.) Liebl.) and Beech (*Fagus sylvatica* L.) were used, from the highland near Sopron, Hungary. The dimension of the samples is always 200x20x20mm^3 (L x T x R), adapting to the compressing machine's capacity. For the longitudinal compression an excellent hardwood quality is needed: precise sized, knot- and defects-free, high moisture content and minimal fiber slope, free from cracks and deformations (Báder and Németh 2016).

After the steaming at atmospheric pressure, the samples were accordionisated by 20%. This is a semi-closed, tempered, unique laboratory machine. In the Table 1 the used test methods can be seen for oak wood specie.

Table 1

Test methods and markings of the oak samples

Marking	Explanation
OC	Control
OSC	Steamed Control
O0m	Compressed without relaxation
O1m	Compressed with 1 minute relaxation
O3m	Compressed with 3 minutes relaxation
O5m	Compressed with 5 minutes relaxation
OLm	Compressed with a long-time relaxation

During the relaxation phase the pressing force is decreasing. In the first minute about with 30%, and the decreasing gradually slows down but the process still goes on even after a day. At the *OLm* samples the heating of the laboratory compression chamber was switched off, so the samples did not lose much from their original humidity content.

Earlier investigations (Báder and Németh 2016) showed that unequal compression rate along the length befall very rare and in most cases if a mistake happens, the result is obvious, so the equal compression rate proof was not necessary at this investigation. Two cracked pieces of more than hundred samples were found, these were kept out from the next measurements.

Bending test

Before the tests were made (bending test and nanoindentation), the control- and accordionisated samples were stored at normal condition (20°C / 65%) until they reached the equilibrium moisture content.

The samples thickness (*h*) had to be cut back to 13,0mm, to provide the proper support-span/specimen-thickness ratio (*L/h*) for the 4 point bending tests. In the Equation 1 the determination of the *MoR* can be seen, while the *MoE* and the maximal deflection (y_{max}) were determined with the Equation 2 and 3 by Kossa (2013).

$$MoR = \frac{3 \cdot F \cdot a}{b \cdot h^2} \; [MPa] \qquad (1)$$

where *F - maximum load, in N;*
a - distance between the loading position and the nearest support, in mm;
b - width of the sample's cross section, in mm.
h - thickness of the sample's cross section, in mm.

$$MoE = \frac{\Delta F \cdot a^2 \cdot (3 \cdot L - 4 \cdot a)}{12 \cdot I_x \cdot \Delta w \cdot 1000} [GPa] \quad (2)$$

where ΔF - *difference between the 10% and 25% of the maximum load, in N*
L - *distance between support span pins, in mm*
I_x - *second moment of area, in* mm^4
Δw - *increment of the load span displacement corresponding to ΔF, in mm.*

$$y_{max} = \frac{F \cdot a \cdot (3 \cdot L^2 - 4 \cdot a^2)}{48 \cdot I_x \cdot MoE_y \cdot 1000} [mm] \quad (3)$$

where MoE_y - *bending elasticity moduli belongs to the measured bending force, in GPa.*
Equation (3) was originally specified to get the y_{max} for low deflections, but our samples had high ones. With having numerically the real deflection of some samples during the bending test, we could obtain a linear supplemental equation (Equation 4):

$$y_{max\,real} = 1{,}1563 \cdot y_{max} - 0{,}7345 \; [mm] \quad (4)$$

where $y_{max\,real}$ - *real maximum deflection of the sample, in mm.*

During bending the annual rings' position was in standing direction. The bending test of the control samples was made with 8mm/min load span displacement speed, while the speed at the compressed samples was 16mm/min, considering the greater deflection based on the Hungarian standard MSZ 6786-5:1976.

The specimens were dried in a 103°C temperature oven to 0% moisture content after the bending test. Owing to the store in normal conditions, the moisture contents were all the same, about 14% at the time of the bending examinations. We used the general conversion method Equation (5) for the comparable mechanical properties.

$$\sigma_{12} = \sigma_u \cdot [1 + \alpha \cdot (u - 12)] \quad (5)$$

where σ - *measured mechanical property (in this case MoR and MoE)*
α - *a constant hanging on the type of the investigated mechanical property*
u - *moisture content, in %.*

α=0,04 for the *MoR* and α=0,02 for the *MoE* (Kollmann 1951).

Nanoindentation

The small samples for the measurements were made from the middle part of the original samples. The originals were the *OC, OSC, O0m, O1m, O3m, OLm* oak, and *BC, BSC, B0m* beech samples. About 2x2mm^2 end grain surfaces were made, from the same annual ring, the latewood section. The small samples were embedded in epoxy resin by alternating vacuum-pressure. They were dried at 60°C, then parallel surfaces were cut and glued to metal plates, perpendicular to the grain. A smooth surface was made for all samples with a Leica Ultracut-R microtome equipped with Trim 45° and Histo diamond knifes (Diatome, Switzerland). All moisture contents were about 9-10% at time of the embedding.

The NI experiments were performed with a Hysitron Triboindenter® (Minneapolis, USA) and a Berkovich-type indenter tip. The mechanical properties of the S2 cell wall layer were measured. The device imprints the tip in the selected point of the cell wall and measures the properties of the operation. The measured cell wall has to at least 3µm be thick. For example the indentation modulus and the hardness of the cell wall can be obtained this way, using the Oliver and Pharr method (1992).

In each indentation process the tip reached the sample surface by a preforce of 2µN, then followed the test by three loading segments, as it is shown on the Fig. 2.

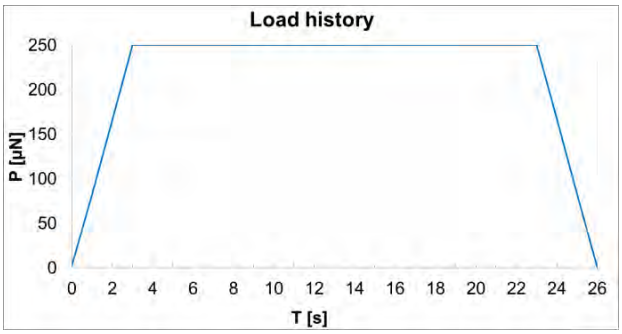

Fig. 2.
Nanoindentation load history.

The peak load (P_{max}) and the contact area (A) are recorded during the experiment. By dividing P_{max} by A, hardness (H) could be calculated. The indentation modulus (E_r) is determined from the initial slope of the unloading curve (Konnerth and Gindl 2006). E_r takes into account the indenter tip's compliance. The influence of the diamond indenter's indentation modulus is negligible.

For NI the TriboScan v8.2.0.18 (Hysitron Inc, Minneapolis, USA) software was used. The 3D pictures were made with TriboView (Hysitron Inc, Minneapolis, USA) software. A typical example can be seen on the Fig. 3 for the appropriate NI measurements on a 15x15 µm segment, with the imprints in the middle of the thick wood cell wall around the lumen.

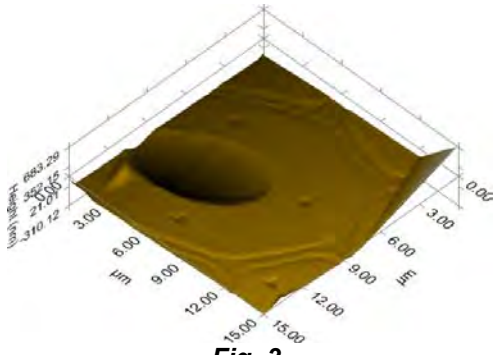

Fig. 3.
A nanoindented segment.

RESULTS AND DISCUSSION

Macromechanical changes

Four point bending tests were carried out as described above. The differences between the control and the modified samples' mechanical properties are shown in Table 2.

Table 2

Changes in mechanical properties of the oak material during the modification, compared to the control samples. Abbreviations: OC-control sample; OSC-steamed control sample; O0m, O1m, O3m, O5m-longitudinally compressed samples with 0, 1, 3 and 5 minutes relaxation time; OLm-compressed and long-time relaxed sample

Treatment name	Stress at 5 mm load span displace-ment	MoR	MoE
OC	100,0%	100,0%	100,0%
OSC	101,5%	102,2%	100,8%
O0m	46,8%	96,8%	41,5%
O1m	43,9%	95,4%	37,0%
O3m	41,5%	94,0%	34,2%
O5m	40,2%	93,1%	31,4%
OLm	29,3%	48,5%	18,6%

Between *OC* and *OSC* samples the differences are negligible. It can be clearly seen, that the stress at 5 mm load span displacement and the *MoE* decrease to less than half of the original values by the accordionisation. With the relaxation time these two properties are decreasing further. However, *MoR* behave differently, just a long-time relaxation can cause a remarkable decrease. The long-time relaxation has an advantage, too. The *OLm* samples did not break in the course of the bending test. They could tolerate the highest deflection without breaking, this means their deflection ability is at least 6 times higher compared the deflection ability of control samples, while the accordionisated and the short-relaxed samples maximum deflection is 3-4 times higher than the control samples' deflection till the break. The same bending rate needs much lower force by the accordionisation and the increasing relaxation time.

The change of the compressing stress during the relaxation is the highest in the first minute, more than 30% (Fig. 4). Leaving the first period the deflection of the compressing stress slows down, but does not stop. *OLm* samples lost about 75% of their original compressing stress.

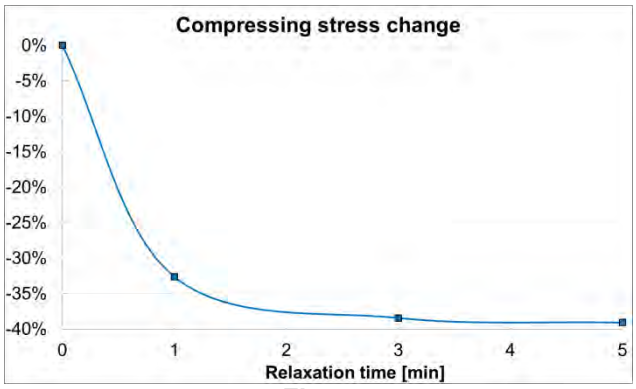

Fig. 4.
Change of the compressing stress in oak wood during relaxation.

Some physical and mechanical properties follow the behaviour of the compressing stress. *MoE*, stress at 5 mm load span displacement and remaining length change are decreasing similarly, but does not have a great extent. This can be also seen in Table 2.

For the easier and higher deflection mostly the decreasing of MoE is responsible (Divos and Tanaka 2005, Wood 2011). The maximum deflection abilities can be seen for the different relaxed samples on the Fig. 5.

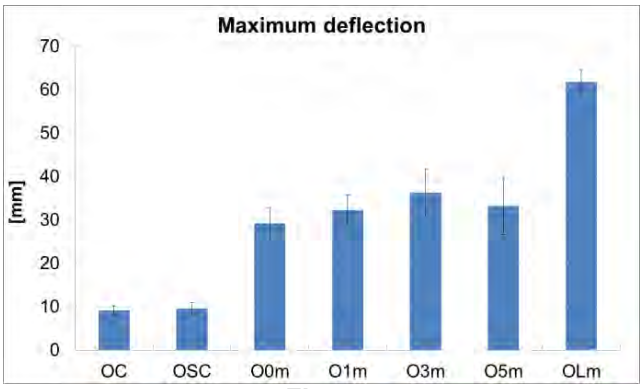

Fig. 5.
Maximum deflections for the control and the modified materials. Abbreviations: OC-control sample; OSC-steamed control sample; O0m, O1m, O3m, O5m-longitudinally compressed samples with 0, 1, 3 and 5 minutes relaxation time; OLm-compressed and long-time relaxed sample.

The maximal deflection of *OC* and *OSC* control samples is similarly. With the accordionisation the deflection multiplies, and finally the *OLm* group shows much more higher deflection, compared to the control materials. The decrease of *O5m* samples deflection compared to *O3m* can be because of the lower sample number and the lower sample quality. However, this difference does not show a significant discrepancy. The ratios of the deflection (Fig. 5) are similar to the *MoE* in Table 2.

Micromechanical changes

On Fig. 6 can be seen well the difference between the treatments effects. While the steaming doesn't resulted great changes in the wood cell's indentation modulus, the accordionisation reduces E_r appreciably, with 25,0% for the oak and 21,0% for the beech cell walls. Short-time relaxations resulted in marginal changes in the E_r. However, long-time relaxation led to a great reduction again, by 34,8% more, according to the *O0m* group.

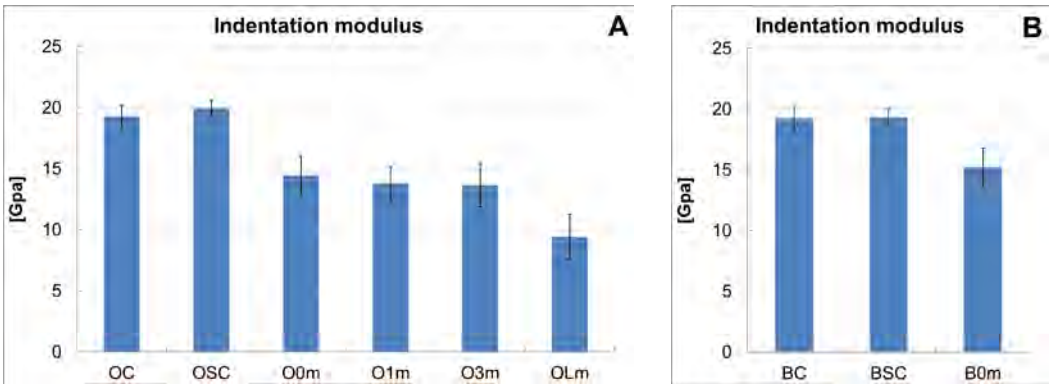

Fig. 6.

Indentation moduli of the control- and differently treated samples: A – oak; B – beech. Abbreviations: OC and BC-control sample; OSC and BSC-steamed control sample; O0m and B0m, O1m, O3m-longitudinally compressed samples with 0, 1 and 3 minutes relaxation time; OLm-compressed and long-time relaxed sample.

Fig. 7 shows the changes in the same direction for *H*. The hardness of the cell walls also decreasing with the accordionisation and with the relaxation time, but in smaller rate than the E_r. The difference between *OC* and *O0m* groups is about 6,4% for the oak and 16,8% for the beech cell walls, and between *O0m* and *OLm* groups are 6,2% again. We can state, that hardness does not reduce as considerably as the E_r.

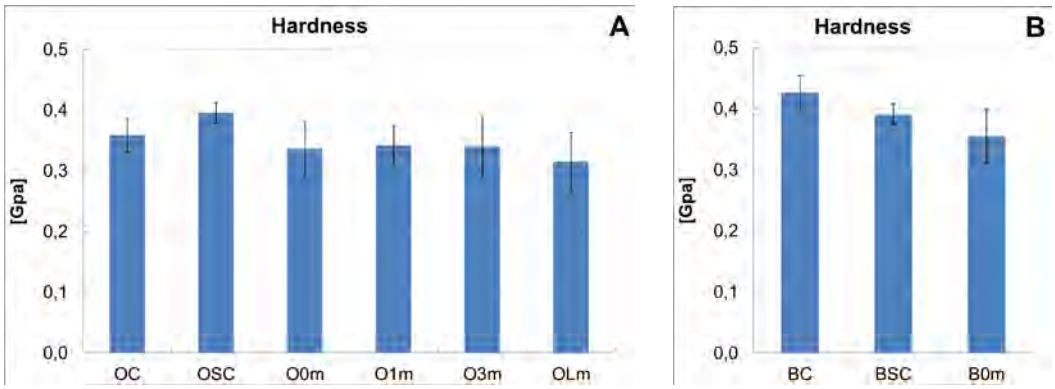

Fig. 7.

Hardness of the control- and differently treated samples: A – oak; B – beech. Abbreviations: OC and BC-control sample; OSC and BSC-steamed control sample; O0m and B0m, O1m, O3m-longitudinally compressed samples with 0, 1 and 3 minutes relaxation time; OLm-compressed and long-time relaxed sample.

It can be concluded that due to this mechanical modification the structure of the cells change. In the bendability the elastic modulus plays the most important role. On the cellular level the indentation modulus deflects greatly (*OC-O0m* 25,0% and *OC-OLm* 59,8%), but not so much as *MoE* on the macroscopic level (*OC-O0m* 60% and *OC-OLm* 80%).

CONCLUSIONS

The purpose of the wood's longitudinal compression is to make the wood bendable. This study was performed to specify the changes of some main mechanical properties such as *MoE* and some physical properties such as deflection of Sessile Oak, by the accordionisation and the different relaxation times.

MoR does not change remarkable by the modification, except at the long-time relaxation it deflects to the half of the original value. However, stress at 5 mm load span displacement deflects with 53,2% because of the accordionisation and 70,7% because of a long-time relaxation. So bending to the same radius of an accordionisated, and even more a long-time relaxed wood needs much lower force. These values in the case of *MoE* are 58,5% and 81,4%, respectively. The latter fact explains the excellent bendability of this type of modified wood. On the cellular level also can be seen great changes in the value of indentation modulus, but not so much as on the macroscopic level (25,0% for the accordionisation and 59,8% for the long-time relaxation). The cell wall's hardness also decreases. Compression rate and relaxation time both significantly affect the mechanical properties of accordionisated wood.

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SUITABILITY OF WOOD OF NATIVE OAK SPECIES (*Quercus spp.*) FROM THE IBERIAN PENINSULA NORTHWEST FOR MANUFACTURE OF BARRELS FOR WINE AGING

Ignacio J. DIAZ-MAROTO
University of Santiago de Compostela, Higher Polytechnic School
Campus Universitario s/n, 27002 Lugo, Spain
Tel: 0034 982 107531, Fax: 0034 982 823001, E-mail: ignacio.diazmaroto@usc.es

Pablo VILA-LAMEIRO
University of Santiago de Compostela, Higher Polytechnic School
Campus Universitario s/n, 27002 Lugo, Spain
E-mail: pablo.vila.lameiro@usc.es

Abstract

The difficulty of our research was to study the characteristics and/or properties of wood of native oak species for the manufacture of wine barrels. An inventory has been made in the oak forests of the most abundant species of the genus Quercus in the Northwest of the Iberian Peninsula, in general, and in the Galician region, particularly. Subsequently, we have calculated the essential parameters of the wood, using samples of different trees which have been felled in the inventoried forests to assess the suitability of oak wood for manufacturing of barrels. Initially, this will allow us to even propose in advance whether or not to carry out certain silvicultural treatments to achieve our goal. The overall objective was to make a description of the most important properties of wood of these species for their possible use in the cooperage industry. The current study about wood properties of Quercus robur and Q. pyrenaica is based on the determination of bark, sapwood, and heartwood to estimate their variation in the Galician oaks. Quercus robur has a higher proportion of bark (22.9%) than Quercus pyrenaica (17.6%), but the high coefficient of variation associated with this value (62.7%) moderate this statement.

Key words: *oak wood; oak barrel; wood properties; cooperage industry; Galicia.*

INTRODUCTION

With 18.2 million hectares of forest cover, Spain is the third country in Europe in terms of forest resources (following Sweden and Finland, but excluding the Russian Federation). Forests occupy almost 29% of the country total area, are increasing by about 116,000 ha per year. Forest fires are a major problem, although there is large variation from year to year. In 2015, forest fires affected 102,946 hectares (0.37% of the Spanish forest area) (MAGRAMA 2015).

From an economic viewpoint, the contribution of the forestry sector is strategic for Galicia; the Galician forest area is 2 million hectares, representing the 69% of the total geographical area. Galicia has almost 11% of the Spanish forest area, which is a sign of extraordinary importance and scale of the forestry sector (MAGRAMA 2011). Most forests are concentrated in the Lugo province, which has a 34% of the total. Oak forests, pure stands or mixed with other deciduous (ash, birch, cherry, chestnut, hazel, maple, rowan, and even beech, in areas with higher altitudes), occupy an area of 246,445 ha, i.e. 18% of the Galician forestry area (MAGRAMA 2011). These species have a robust temperament and are light-demanding, which does not tolerate shade at first stages of development and the seedlings languishing quickly undercover. The most important oak stands are found on steep slopes where have survived because felling would be very difficult (Ruiz de la Torre 1991). The most abundant species is *Quercus robur* L., followed by *Quercus pyrenaica* Willd., with scarce presence of *Quercus petraea* Liebl., whose stands are mainly inhabit in mountainous areas with complicated topography and its access prevents use of forestry machinery. Therefore, we have not included the inventory and assess of the *Quercus petraea* stands in our study.

The oak trees are little used in the forestry industry, the small plots and large number of owners doesn't facilitate its forestry, timber exploitation, as well as the later industrial development. Forest management of oak forests is virtually nonexistent and the use of oak wood is limited for firewood (Diaz-Maroto et al. 2005). This is the main reason why it would be well advised to develop an industry for a better value of these species.

Oxidative aging of wine in oak barrels is a traditional practice in wine-growing areas recognized as being production of quality wines (Cutzach et al. 1999). It is currently undergoing expansion and

introduction in many areas, due to the need to improve the quality of wines to compete in an oversupplied market and at the same time becoming more demanding (Martinez 2004).

Galicia has, among others, a wine-growing area called *Ribeira Sacra*. The geography and climate of this special site, its landscape and the quality of the wine produced, have enabled the creation of the Protected Designation of Origin (PDO). Now, Galicia has four other PDOs, *Rias Baixas*, *Ribeiro*, *Monterrei*, and *Valdeorras*. The winegrowers with more wine tradition of the *Ribeira Sacra* are passionate about their region. They abstain to make the aging of wines in French oak barrels or American, because wood aromas aren't produced of Galicia (Alañon et al. 2011a). For that, they use the old French oak barrels in which wood aromas have disappeared a long time, so the wine retains its authentic *Galician bouquet*. However, it hasn't aromas that make the wine unique. A good solution for this dilemma would be to use oak barrels of Galicia; the wine would keep its local origin and benefit of the wood aromas. Therefore, using Galician oaks for making barrels would be a great way to develop a cooperage industry (Alañon et al. 2011b).

OBJECTIVE

Our study about wood properties of *Quercus robur* and *Q. pyrenaica* is based on the determination of bark, sapwood, and heartwood to estimate their variation in the Galician oaks. The overall objective of the present research was to study the wood properties of two species native oaks of Galicia (*Quercus robur* and *Q. pyrenaica*) for the manufacture of wine barrels. Our aim was to carry out a description of the most important properties of wood of these species for their possible use in the cooperage industry as key for sustainable development in many rural areas.

MATERIAL AND DESCRIPTION OF THE METHOD

The study area, Autonomous Community of Galicia (Fig. 1), was considered as a single unit where zones for data collection were selected taking care to include a suitable representative number of oak stands, on the basis of the Forest Map of Spain (Ruiz de la Torre 1991). The minimum area of the stands ranged between 0.5 and 1 ha, which avoided problems related with the edge effect. The current study about wood properties of *Quercus robur* and Q. *pyrenaica* is based on the determination of sapwood and heartwood to estimate their variation.

For that, it was necessary to fell several trees within the study area. In total, 30 trees were chosen in 11 different stands of the provinces of Lugo and Ourense, i.e. we have obtained 30 wood slices of *Quercus* at 60cm tall on the trunk of the tree. The wooden material used consisted in 142 wood samples with a parallelepiped shape and dimensions of 20x20x40±1mm (Fig. 2). We can notice that the accuracy of ±1mm is not really important because we can overcome the inaccuracies of the geometric shape thanks to the water displacement method.

As 69% of Galicia is over 600m above sea level, that's why a lot of stands are located on zones with steep slopes. Since as we mentioned above, the most important oak forests are found on abrupt sites, where they have survived because felling would be complicated owing to the topography. This fact is a problem because a strong slope encourages the creation of the tension wood with a heart off centre, which is not appropriate to manufacture barrels (Lehringer et al. 2008).

Fig. 1.
Study area.

Fig. 2.
Wood samples with a parallelepided shape.

Table 1 presents the main characteristics of the slices of wood from trees felled in the study area (from normal tree to heart strongly off centre).

Table 1

Main characteristics of the slices of wood from trees felled

Species	Normal tree	Hear a little off centre	Hear off centre	Hear strongly off centre	Total
Quercus robur	7	4	4	2	17
Quercus pyrenaica	5	2	3	3	13
Total	12	6	7	5	30

To determine the width of each of the parts of the trunk (bark, sapwood and heartwood) on the wood slices would be sufficient to use a ruler with an appreciation of a millimetre. However, in our case we have worked with the *ImageJ* software (Fig. 3), which has greatly facilitated us the high number of meticulous measurements to be made. *ImageJ* is public domain Java software for image processing. With it the whole sample wood will be used and the singularity of each sample will be considered. Also, we can calculate the area of each part of the tree and their relative proportion on the wood slices. For that, it is necessary to use the polygon selection tool to create irregularly shaped selections defined by a series of lines. To generate a polygon selection, we need to click repeatedly with the mouse to create line segments that correspond to the border of the various areas. *ImageJ* has an automatic selection tool called Wand Toll. We can used the tolerance of this tool to define the border of the automatically selection. The determination of the bark border is the most difficult. The perimeter is irregular, and sometimes it's difficult to see clearly the limit of the border. The area of the sapwood could be calculated by subtraction of the heartwood area. Finally, the area of the bark could be calculated by subtraction of the sapwood area. *ImageJ* is multithreaded, so time-consuming operations such as image file reading can be done in parallel with other. It supports standard image processing functions such as contrast manipulation, sharpening, smoothing, edge detection and median filtering. Spatial calibration is available to provide real world dimensional measurements in units such millimetres (Ferreira and Rasband 2012).

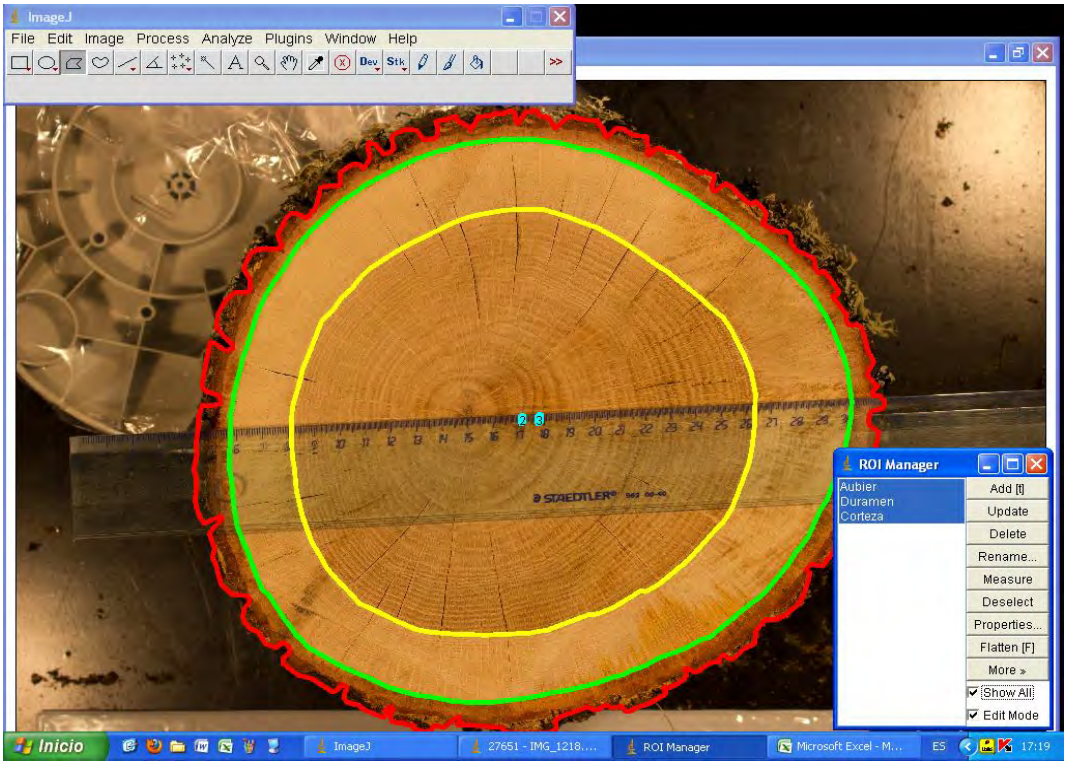

Fig. 3.
Area of the heartwood (yellow line), sapwood (green line) and bark (red line).

The photographs were taken by a Canon EOS 550D. This camera uses a special structure that ensures an optimal light and the distance between the camera and samples is always the same. The precision of the photographs is optimal. The first step consists to define the spatial scale of the active image so measurements results can be presented in calibrated units. In this step, the centimetre unit will be used. Before using this command, we need to make a line selection that corresponds to a known distance. *ImageJ* will have automatically filled in the distance in pixels based on the length of the line selection (Ferreira and Rasband 2012).

RESULTS AND DISCUSSION

The selection program of the samples follows the criteria following:

- Two species of oak: *Quercus robur* and *Quercus pyrenaica*
- Three types of wood: sapwood, heartwood, and juvenile wood
- Three types of tree: with rotten heart, with an off-centre heart, and normal tree

Table 2 shows the distribution of the wood samples in function of the type of wood (sapwood, heartwood, and juvenile wood) and the species.

Table 2

Distribution of the samples depending of the species and the wood type

Wood type / Species	***Quercus robur***	***Quercus pyrenaica***	**Total**
Sapwood	33	29	62
Heartwood	27	34	61
Juvenile wood	8	11	19
Total	68	74	142

Table 3 presents the distribution of the wood samples in function of the type of tree (from tree with rotten heart to normal tree) and the species. As we can see, no slice of wood has rotted heart.

Table 3

Distribution of the samples depending of the species and the tree type

Tree type / Species	***Quercus robur***	***Quercus pyrenaica***	**Total**
Tree with rotten heart	0	0	0
Tree with an off-centre heart	19	16	35
Normal tree	49	58	107
Total	68	74	142

Table 4 shows the measurements of the area of bark, sapwood and heartwood for each species. The average age of the trees is also shown to facilitate global interpretation. *Quercus robur* has a higher proportion of bark (22.9%) than *Quercus pyrenaica* (17.6%), but the high coefficient of variation (CV) associated with this value (62.7%) moderate this assertion (Table 4). Also, as expected, the accuracy of bark measurements is relatively low because of its extreme irregularities. Age does not appear, in this case, have a significant influence neither in the proportion of bark nor in the sapwood, but if in the proportion of heartwood. This situation does not mean that there is not link between sapwood proportion and age (Lévy et al. 1992; Lebourgeois et al. 2004; Pasztory et al. 2014); the only conclusion is that our data are not able to reveal this link. Probably, the variation of the values is too high: the coefficient of variation are superior to 30% for the two species –*Quercus robur* (32.1%) and *Q. pyrenaica* (30.5%)– (Table 4), since our range of age is probably too small for *Quercus pyrenaica* (only 30 to 64 years) (Fernandez-Parajes et al. 2005). Finally, we note that the accuracy of the heartwood area for *Quercus pyrenaica* is less accurate than expected (> 1%). This situation can be explained by the presence of heartwood area without accurate limits. This is not the case on the wood slices of *Quercus robur* (Vivas 2000; Pot et al. 2013).

Table 4

Area of the bark, sapwood and heartwood for each species and average age in function of the species and the wood type

Species	Number of trees	Type of wood	Average (%)	Standard deviation (%)	Coefficient of variation (%)	Accuracy (± / %)	Minimum (%)	Maximum (%)	Average age
Quercus robur	17	Bark	22.9	14.3	62.7	1.4	12.0	30.3	55
		Sapwood	41.4	13.3	32.1	1.6	26.9	69.3	55
		Heartwood	38.3	11.1	29.0	0.8	18.2	59.0	55
Quercus pyrenaica	13	Bark	17.6	3.7	20.9	1.5	9.8	29.0	48
		Sapwood	44.7	13.6	30.5	0.7	12.9	60.6	48
		Heartwood	37.7	14.6	38.8	1.3	11.7	70.1	48

CONCLUSIONS

The assessment of the different proportions of bark, sapwood and heartwood of the Galician oaks and its relation with the age has allowed knowing the following aspects:

- The proportion of heartwood is positively proportional to the age of the tree.
- When a tree grows quickly the proportion of heartwood is higher at the same age than in other trees.
- The rapid growth of *Quercus pyrenaica* causes the formation of large proportion of heartwood in a few years.
- The heartwood proportion is an average between 50.2 and 53.6% (maximum and minimum values of the average ± standard deviation ± accuracy).
- On the contrary, data from this study do not make it possible to respond to the existence of a relationship between age and proportion of bark and sapwood.

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SELECTED CHARACTERISTICS OF NORWAY SPRUCE WITH INTENDED RINGS (HAZEL GROWTH) FOR VIOLINS

Anton KRALER
University of Innsbruck, Institute for Structural Engineering and Materials Science
Technikerstr. 13, 6020 Innsbruck, Austria
Tel: 0043 512- 507 63206, Fax: 0043 512-507 63299, E-mail: anton.kraler@uibk.ac.at

Wilfried BEIKIRCHER
University of Innsbruck, Institute for Structural Engineering and Materials Science
Technikerstr. 13, 6020 Innsbruck, Austria
E-mail: wilfried.beikircher@uibk.ac.at

Armin ROFNER
University of Innsbruck, Institute for Structural Engineering and Materials Science
Technikerstr. 13, 6020 Innsbruck, Austria
E-mail: armin.rofner@uibk.ac.at

Abstract

The paper presents the results of an experimental research performed with spruce wood (Picea abies L.) with indented rings (hazel growth), originated from the same forest parcel from the Pitztal valley in Tyrol, Austria. The material was harvested at a sea level ranging from 1450 to 1700 m and the wood was air dried for 2-9 years. The supplier evaluated and sold the resonance wood samples at the highest quality grades for violin and/or viola decks. The results showed that the density of 0.407 g/cm³ and the ultrasonic velocities of 5684.47 m/s in longitudinal, 2174.08 m/s in radial and 1546.66 m/s in tangential direction are in the range of high quality resonance wood. But the shear strength of 9.63 N/mm² in the longitudinal-tangential direction and 9.56 N/mm² in the longitudinal-radial direction shows very high values in comparison to literature data of 6.7 N/mm². The overall conclusion of this research is that Norway spruce with indented rings selected as resonance wood shows high potential to be used for string instruments, but because of the high shear strength in longitudinal direction, it is more difficult to carve than "standard" spruce resonance wood.

Key words: *spruce; hazel growth; tone wood; acoustical properties; modulus of elasticity.*

INTRODUCTION

The superior sound of some instruments has remained a mystery up to now. On the one hand the delightful sound of famous violins of e.g Stradivari or Guarneri (del Gesú) can be explained by the high quality of the construction and workmanship of these instruments. On the other hand there is no doubt that the selection and quality of the raw material plays an important role (Sacconi 1977).

The different parts of a violin are traditionally made from different types of wood: ebony and rosewood for the fingerboard, maple for the bridge, and spruce for the soundboard of the body (Sacconi 1977). The soundboard amplifies the resonance of the strings and is highly responsible for the tonal qualities of the instruments (Buksnowitz 2006). Opinions among instrument makers as to which resonance wood should be used differs. Some prefer Norway spruce with the grown pattern of indented rings (hazel growth), and others without this feature.

Although the hazel-growth doesn't have perfect aesthetic properties and is often seen as a growth defect, many experts perceive the hazel spruce for instruments as a sound-improving property (Buksnowitz 2006). The research question involves investigating if the quality of resonance wood can be tested objectively in the laboratory.

The present research focuses on the mechanical and, as far as possible, on the acoustical properties of the hazel. For this purpose, a detailed experimental set-up and the specimens from available material were prepared. The objectives of the investigations, the material used, the method used and the measuring instruments used are described below.

OBJECTIVE

The main objective of the present research was to determine relevant properties from Norway Spruce with indented rings, which is selected as high quality tone wood for string instruments. The determined properties are compared to literature data of „standard" spruce and tone wood to extend

the knowledge whether this specific grown pattern of indented rings constitutes any benefits when used as tone wood. The results of these investigations should also help to increase the value and acceptance of hazel-spruce as a special tone wood.

MATERIAL, METHOD, EQUIPMENT

Specimens for the characterization of mechanical and acoustical properties were obtained from 12 wedge-shaped spruce samples, which the supplier considered to be of the highest quality grades for violin and/or viola decks. The length of the raw samples were between 50 and 95 cm. The provenance was the Pitztal valley in Tyrol, Austria at a sea level ranging from 1450 to 1700 m. The wood was air dried for 2-9 years.

For the determination of the properties defect free samples were produced after storing the wedge-shaped samples until the equilibrium moisture was reached. For the modulus of elasticity (MOE) and sound velocity in longitudinal direction, samples with 20 x 20 x 360 mm³ were prepared. Parts of these samples with the dimension of 20 x 20 x 20 mm³ were used for the determination of moisture and density values. For the determination of the MOE and sound velocity in radial direction, the samples had the size 20 x 200 (180 or 120) x 40 mm³. For the shear strength in longitudinal-radial and longitudinal-tangential direction and sound velocity in all three directions, samples with 50 x 50 x 50 mm³ were prepared. The schematic material preparation is presented in Fig. 1 and Fig. 2.

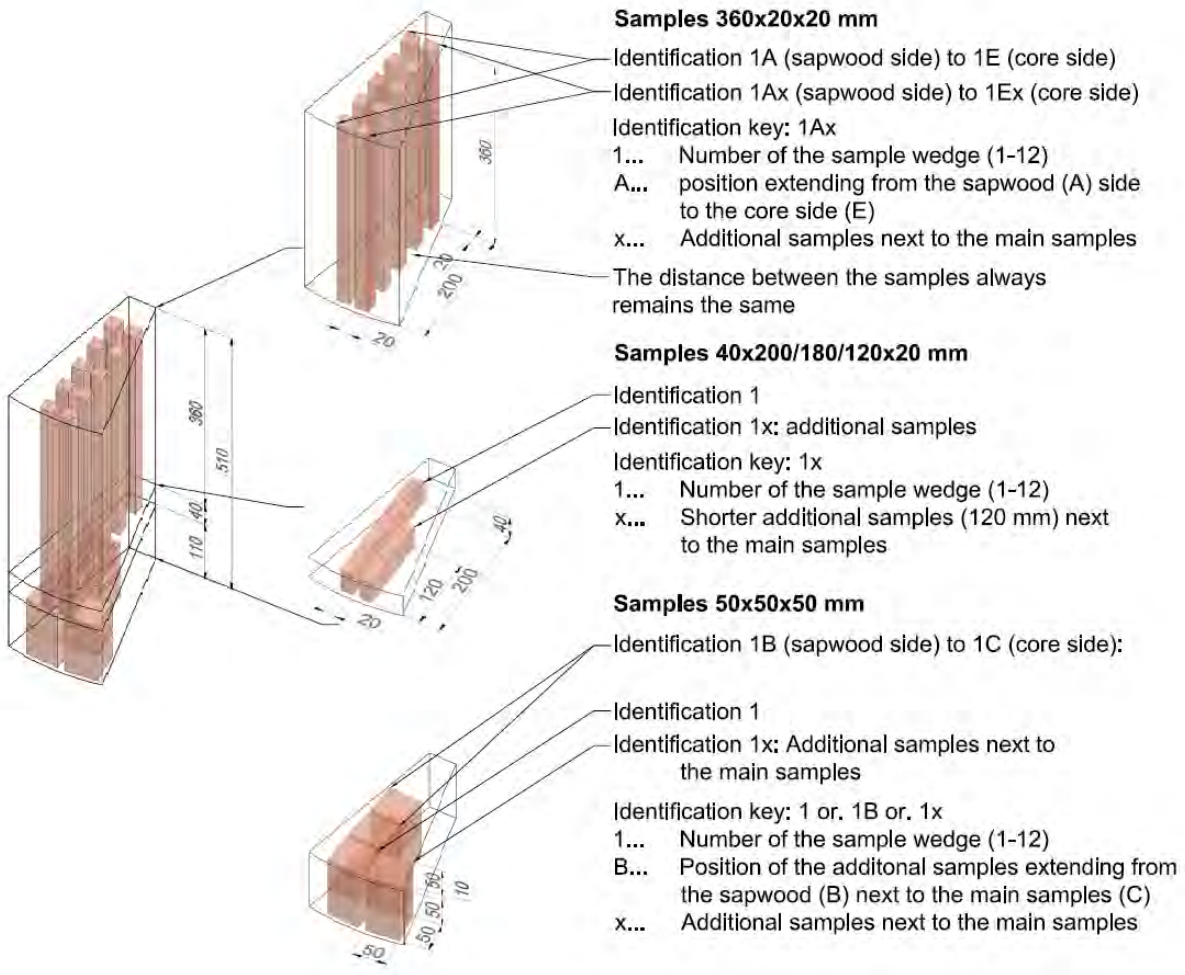

Fig. 1.
Schematic description of the material preparation.

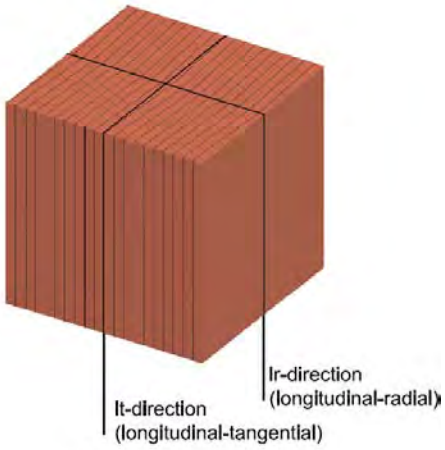

Fig. 2.
Schematic demonstration of the sample for the determination of the shear strength in longitudinal-radial (lr) and longitudinal-tangential (lt) direction.

The determination of the material properties was performed according to the standards for small and defect-free wood samples. The determined material properties were: density, moisture content, modulus of elasticity and shear strength. The specific standards, the number of specimens used for each mechanical test, as well as the devices and some remarks are presented in Table 1. For the determination of the MOE in radial direction, the sample size was altered from the standard to 20 x 200 (180 and 120) x 40 mm³. Therefore the free span was 10 mm shorter than the sample with 190, 170 or 110 mm.

The material tests were performed at the TVFA (Technische Versuchs- und Forschungsanstalt) at the University of Innsbruck. All tests were performed after storing the material in a climate chamber at 20 °C and 65 % RH until the equilibrium moisture content (EMC) was reached.

Table 1
Details concerning standards followed, number of samples for each test and the devices used

Test	Standard	No. samples	Devices	Remarks
density	ISO 3131	90	digital measuring slide, balance	balance accurateness 0.001g
moisture content	ISO 3130	90	balance	accurateness 0.001g
three-point bending test	DIN 52185 (1976)	90 longitudinal and 26 radial	Shimadzu Autograph AG-100kN Testing Machine	cross head speed 7 mm/min
shear strength	DIN 52187 (1979)	26 lr and 26 lt	Shimadzu Autograph AG-100kN Testing Machine	test duration 90 ± 30s

The shear strength was determined in longitudinal-radial (lr) and longitudinal-tangential (lt) direction.

Fig. 3.
Test facilities for the determination of the shear strength in longitudinal-radial (lr) and longitudinal-tangential (lt) direction according to DIN 52187.

The sound transmission with longitudinal waves at the frequency of 220 kHz was performed with the ultrasound measuring device Proceq Pundit Lab by using the transceiver S9204A of the company Physical Acoustic Group (Fig. 4).

Fig. 4.
Ultrasound measuring device Proceq Pundit Lab.

RESULTS AND DISCUSSION

Moisture content and density

The mean value of the moisture content was 13.9 % and the standard deviation was 1.47 %. The mean value of the density was 0.407 g/cm³. The range was from 0.351 g/cm³ to 0.473 g/cm³. The results are given in Table 2.

Table 2

Moisture content and density of the material tested

property	no. samples	mean value	standard deviation	coefficient of variation
moisture content	90	13,87 %	1,47 %	10,6 %
density	90	0,407 g/cm³	0,024 g/cm³	5,8 %

Modulus of elasticity

The modulus of elasticity in longitudinal direction was determined by having the force in the tangential year ring direction as usually required by the standard DIN 52186 and in the radial direction deviating from the standard. The same samples were used for both tests, because the modulus or rupture were not determined and the tests were stopped when the calculated maximum strength of 43 % (of the bending strength) was reached. The maximum strength was determined previously with additional samples.

Table 3 shows that the results of the modulus of elasticity, with the force in the tangential direction of the year rings, were about 140 N/mm² lower than in radial direction.

Table 3

Modulus of elasticity

testing direction	no. samples	mean value	standard deviation	coefficient of variation
longitudinal: tangential year ring orientation	90	9558.61 N/mm²	984.35 N/mm²	10.3 %
longitudinal: radial year ring orientation	90	9697.50 N/mm²	1049.01 N/mm²	10.8 %

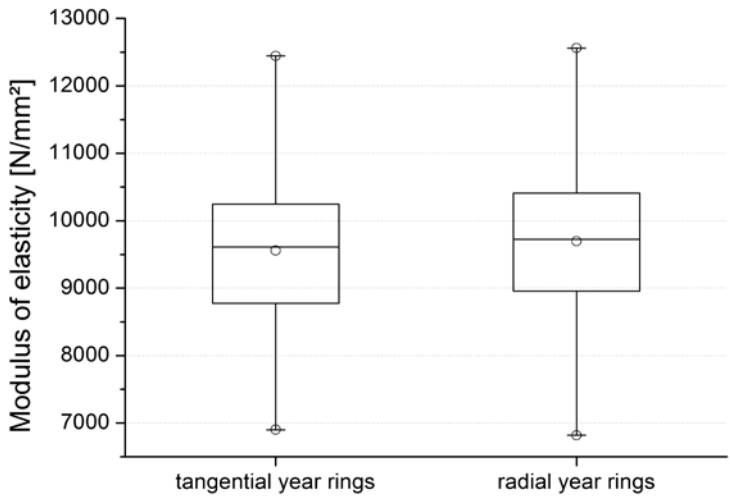

Fig. 5.
Modulus of elasticity in longitudinal and radial direction.

Shear strength

The shear strength was determined in longitudinal-tangential (lt) and longitudinal-radial (lr) direction. The mean value in lt-direction was 9.63 N/mm² and in lr-direction 9.56 N/mm². Because the testing time was too short, three samples in lt direction and four samples in lr direction were not included in the evaluation.

Table 4

Shear strength

testing direction	no. samples	mean value	standard deviation	coefficient of variation
lt	23	9.63 N/mm²	0.66 N/mm²	6.8 %
lr	22	9.56 N/mm²	1.08 N/mm²	11.3 %

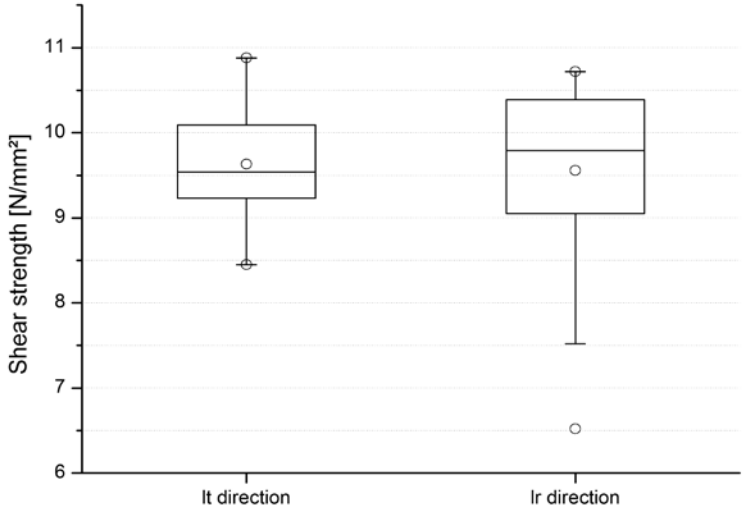

Fig. 6.
Shear strength in longitudinal-tangential (lt) and longitudinal-radial (lr) direction.

The laboratory tests showed that the shear strength of hazel spruce is slightly higher than the shear strength of "standard" grown spruce according to ÖNORM B3012 (6.7 N / mm²).
This can be explained by the larger fibre angle, the interlocking of the annual rings, and the greater proportion of medullary rays of hazel-spruce.

Sound velocity

The sound velocity was measured at 220 kHz in all three directions (longitudinal, radial and tangential) by using samples with 50 x 50 x 50 mm³, in the longitudinal direction by using the samples with the dimension 20 x 20 x 360 mm³ and in the radial direction by using the samples with the dimension 20 x 200 (180, 120) x 40 mm³.

Before the tests, the samples were primed until an equilibrium moisture content of 20 °C and 65 % RH was reached. The transmitter was attached to all samples with constant pressure and no contact medium was used to transmit sound waves from the transmitter to the sample. The sound velocity was calculated according to Eq. 1.

$$\nu = \frac{l}{t} \quad [\text{m/s}] \qquad (1)$$

ν is the sound velocity [m/s], l is the specimen length, path length [m], and t is the transit time of ultrasound [s].

Table 5

Sound velocity of the cube sample 50 x 50 x 50 mm³

testing direction	no. samples	mean value	standard deviation	coefficient of variation
longitudinal	23	5684.57 m/s	187.74 m/s	3.3 %
radial	23	2174.08 m/s	97.48 m/s	4.5 %
tangential	23	1546.66 m/s	90.88 m/s	5.9 %

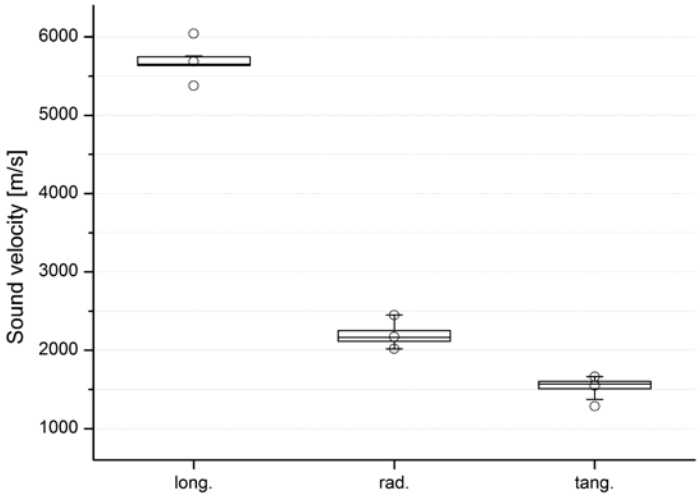

Fig. 7.
Sound velocity in longitudinal, radial and tangential direction.

Table 6

Sound velocity from the bar samples with 20 x 20 x 360 mm³ in longitudinal direction and with 20 x 200 (180, 110) x 40 mm³ in radial direction

testing direction	no. samples	mean value	standard deviation	coeffizient of variation
longitudinal	90	5525.17 m/s	1890.31 m/s	3.4 %
radial	22	2051.12 m/s	143.54 m/s	7.0 %

CONCLUSIONS

The results of the present research demonstrate that

- the elasticity of hazel-spruce is similar to that of "standard" spruce.
- the raw density of the hazel-spruce samples was smaller than that of the "standard" spruce samples from various literature sources. The reason could be the high sea level (1450-1700m) at which the samples were harvested.
- the swelling properties of the hazel-spruce are about the same as those of "standard" spruce.
- the shear strength is an essential parameter for building violins, as it provides information about the cleavage and the process of the material. Values show that the shear strength of hazel-spruce, due to the indentations and the increased size of the medullary rays, is approximately twice as high as that of "standard" spruce and is thus difficult to split. The cleavability is slightly higher along the tangential surface than along the radial surface.
- the sound velocity of hazel-spruce is in the longitudinal as well as the radial direction higher than that of "standard" spruce as compared to the literature sources.

- with the dynamic elasticity modulus determined from the sound velocity, no significant difference to "standard" spruce can be seen. The resonance coefficient is slightly higher, which indicates good sound properties of hazel-spruce.
- there is a positive correlation between the sound velocity and the flexural modulus, so that the sound velocity can be used for predicting the elastic and resonant properties e.g. violins.

ACKNOWLEDGEMENT

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APPLICATION OF HIGH FREQUENCY DENSITOMETRY TO DETERMINE WOOD DENSITY AND RING WIDTH OF BEECH TREES

Dr. Dimitrios KOUTSIANITIS
Aristotle University of Thessaloniki, School of Forestry and Natural Environment, Department of Harvesting and Technology of Wood Products, Greece
E-mail: koutsian@for.auth.gr

Professor Ioannis BARBOUTIS
Aristotle University of Thessaloniki, School of Forestry and Natural Environment, Department of Harvesting and Technology of Wood Products, Greece
E-mail: jbarb@for.auth.gr

Abstract

The variations of wood density and annual ring width were examined on different heights of beech trees. In total, 25 stem discs were collected from 5 beech trees, at 1,3m, 5m, 9m, 13m and 17m height from base of the tree. The wood density and ring width measurements were carried out via high frequency densitometry method, using the Lignostation system. The wood density showed variation within beech trees at different growth heights. There was a tendency for reduction of wood density with tree height, but this did not occur in all beech trees. The annual ring width varied within beech trees. In some beech trees, it gradually increased from the top of the tree to a maximum of around 9 m and then reduced to the base. The wood density showed a small variation between beech trees. There were not significant differences on the ring width of stem discs, between 4 beech trees, while the ring width of a beech tree was found to be the highest on every stem height. The results from this study suggest that the HF densitometry method is a promising technique to assess wood density and ring width. This method provides the variation of density and analyzes the ring width, within and in-between trees of the same species.

Key words: *wood density; ring width; lignostation; beech; high frequency densitometry.*

INTRODUCTION

Wood density is an important index of wood quality as it affects many wood properties (hydroscopicity, shrinkage and swelling, mechanical, thermal, electrical, etc.) related to the industrial processing of wood (Tsoumis 1991). Wood density is commonly used to describe the wood quality in the mechanical and chemical wood industry. There are many reasons for this interest in wood density as it is correlated with a number of wood quality properties (Repola 2006). Wood density and ring width are the most commonly used indicators of wood characteristics. Density is a complex of characteristic and is influenced by moisture, structure (width of growth rings, proportion of latewood), extractives and chemical composition. Even more, there is variation within a tree, between trees of the same species and between species (Tsoumis 1991). The average wood density of a stem is affected by a large number of factors such as tree species, geographical location and other environmental factors, site quality, position of the tree in a stand, tree age and size, growth rate and genetic factors (Repola 2006).

Density is determined by the gravimetric method or by other methods. The density of wood and its variability within growth rings may be determined by use of instruments (densitometers), which measure the absorbed radiation in different positions within a growth ring (Tsoumis 1991). High frequency densitometry is a new method, for measuring relative density variations along wood surfaces, utilizing the dielectric properties of wood. It is based on a simple measurement procedure, it allows extremely fast measurements of wood density variations and no protective arrangements are needed (Schinker et al. 2003).

Many studies into the application of high frequency densitometry method to obtain density and its variations on entire tree-rings as well as other parameters (tree-ring width, early and latewood width and density) along wood surfaces of stem discs and increment cores have been performed (Šilinskas et al. 2016, Matisons 2015, Shchupakivskyy et al. 2014, Clauder et al. 2012, Hochreuther et al. 2012, Linke and Beck 2012, Meinardus et al. 2012).

For instance, in a recent study of Shchupakivskyy et al. (2014) the High-Frequency densitometry method was proved to be applicable to localise the changes in density within an annual ring, in contrast to the gravimetric method. Furthermore, Clauder et al. (2012) found that, in contrast to

the Gravimetric Method, the High-Frequency densitometry method allows to differentiate changes in earlywood and latewood density.

OBJECTIVE

Similar research for the application of high frequency densitometry to detect changes on wood density and tree ring widths along wood surfaces, in Greece, is lacking. The objectives of this study were the potential use of high frequency densitometry to investigate the variations of wood density and tree ring width: a) within the stem of trees, b) between trees of the same species.

MATERIAL, METHOD, EQUIPMENT

Five standing beech trees (*Fagus sylvatica*) were chosen for our investigation, from the Aspropotamos forest district of Kalampaka, western-central Greece. Trees were felled during the harvesting operations on June 2016. Then, stem discs were cut on the breast height (1,3m) and on 4 different heights of the trees (5m, 9m, 13m and 17m from tree base). The experimental material in the present research consisted of 25 stem discs (Fir. 1), five from each beech tree. Furthermore, 25 more stem discs were collected from the same heights as previous, for repeat testings in case of sample failure (discolouration, cracks, etc.).

The diameter of the discs ranged between 15 cm to 50 cm, as the maximum length of the measurement machine was 50 cm. All discs were transferred indoors for storage and were air-dried for three months to a moisture content of 12% (Fig. 2). Furthermore, the sample discs were covered with wood dust to prevent discs from cracking, due to moisture content reduction.

The moisture content of the discs was checked with an electronic moisture meter. Furthermore, moisture content was estimated by the following equation

$$MC(\%) = \frac{M_{wet} - M_{dry}}{M_{dry}} * 100 \quad (1)$$

where: M_{wet} is the initial weight;

M_{dry} the oven dry mass of the stem discs.

Fig. 1.
View of the collected beech stem discs.

Fig. 2.
Storage of stem discs for indoor air-dry.

The relative density measurements were carried out via high frequency densitometry. The method is based on the propagation of continuous electromagnetic waves in a high-frequency (HF) transmitter-receiver link of an extremely small electrode system, which is in direct contact with the wood surface investigated (Schinker et al. 2003).

For our experiment, we used the measuring system LignoStation (Fig 3.). LignoStation is used to determine information about relative radial density variances of dry wood samples (stem disks or increment cores) using high-frequency scanning of a probe with a very thin tip. The HF-probe measures the di-electric constant of wood, which is proportional to the spatial density (Rinntech 2006).

Before scanning, the sample surface had to be smooth and plain. This was done with LignoTrim, a milling tool with fly-cutter (Fig. 4). Then, the scan-path had to be scanned optically with LignoScop, a microscope camera. After this step, density variations were measured along the smoothed surfaces, using the high frequency dielectric scanner (Fir. 5).

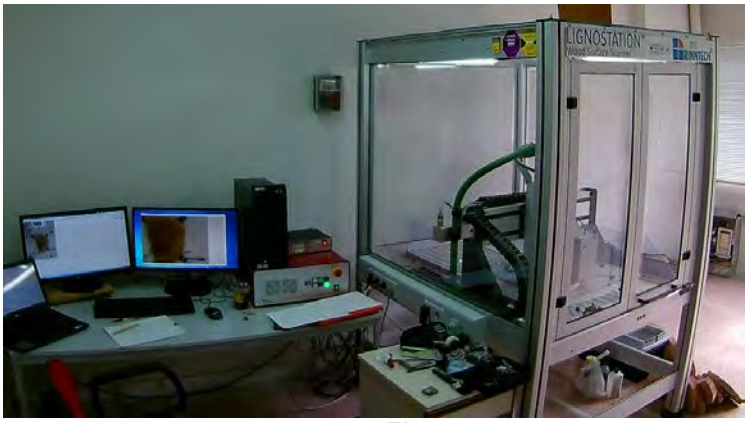

Fig. 3.
The Lignostation machine based in the School of Forestry and Natural Environment, Aristotle University of Thessaloniki, Greece.

Fig. 4.
Disc surface milling with the fly cutter.

Fig. 5.
Disc surface scanning with the high frequency probe.

Prior to density measurements, the measuring system (probe-transmitter-receiver) of the machine was calibrated with a set of standards for wood samples. Next step was to set the milling parameters of the machine. After several trial cuttings, the milling speed, depth and number of paths, were adjusted. The HF probe measurement was highly depended on the wood milling surface quality. It was found that samples of low milling surface quality gave lower values of density measurement, than that of better surface quality. The measurements of the discs were carried out from bark to pith. When any abnormalities in wood surfaces appeared, like knots, cracks, and checks, the milling path was corrected and set again in surfaces without defects.

RESULTS AND DISCUSSION

Table 1 shows the density and moisture content mean values of the selected stem discs calculated from the lignostation system and the oven-dry density values calculated with the gravimetric method.

Table 1

Mean values of density and calculated oven dry density values

Beech trees	Mean density (kgr/m^3)	Moisture content (%)	Oven-dry density (kgr/m^3)
	529,0	12,0	690,5

Certain differences on density values were found to exist among high-frequency densitometry and gravimetric method. These differences may be attributed to the milling surface quality of the stem discs. It was observed that a milling surface of bad quality affects the density measurement with the HF probe, giving lower values of density than the surface of better quality. This variance on density values could be also due to the appearance of defects on the stem discs surface or on the existence of reaction wood. Tsoumis (1991) stated that the density of tension wood can be 2-20% higher than normal wood.

Although proportion of earlywood and latewood, earlywood and latewood density, minimum and maximum density were also determined for every stem disc, only mean stem density values and annual ring width were analyzed in this study. The density and ring width analysis of a beech stem disc, as carried out with the Lignostation software, are shown in Figure 6. The upper part of the graph indicates the variation of density values of the stem disc from bark to pith, as measured by the HF probe. While, the lower part of the graph depicts the ring width measurement of the disc from pith to bark. On the right part of Figure 6, the milling and scanning paths of the stem disc are shown.

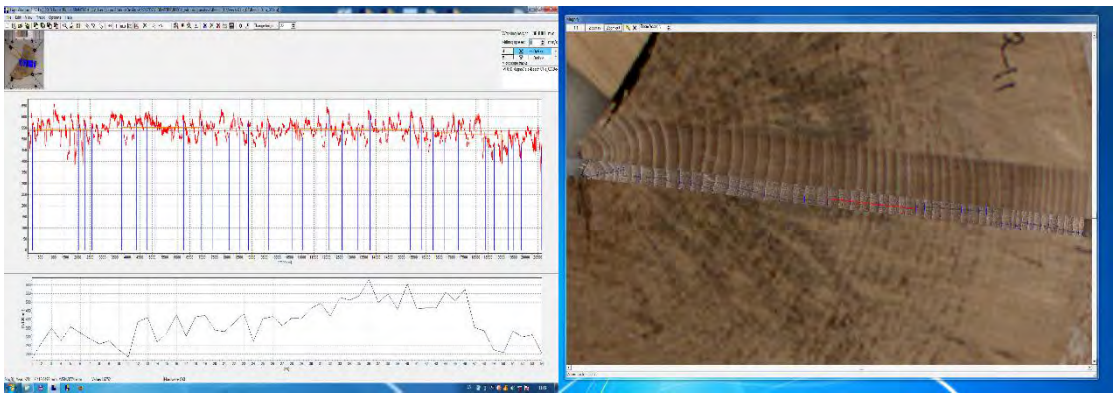

Fig. 6.
The density and ring width analysis on beech stem disc with Lignostation software.

Variation of the wood density and the annual ring width within beech trees

The variation in wood density at different heights are shown in Figure 7. From the base to the top, the highest average wood density for all beech trees was found at breast height (1,3m). There was a tendency for reduction of density with tree height, but this did not occur in all cases. For instance, beech tree 3 showed a constant decrease in density from base to top of the tree. While, beech trees 1, 2 and 4 indicated a reduction in density from the breast height to 9m long height of the tree, then slightly increased with stem height and finally beech trees 1 and 4 declined. Beech tree 5, with the highest density, showed a decline in wood density below 5m stem height. Overall, the average wood density presented a decreasing trend with stem height.

This reduction may be attributed to mechanical factors. Under the influence of weight, wind, and snow on the crown, greater stresses develop at the base of the trunk, resulting in local formation of wood of higher density. Furthermore, greater density at the base of a tree is contributed by the formation of heartwood, as the proportion of heartwood is higher at the base (Tsoumis 1991).

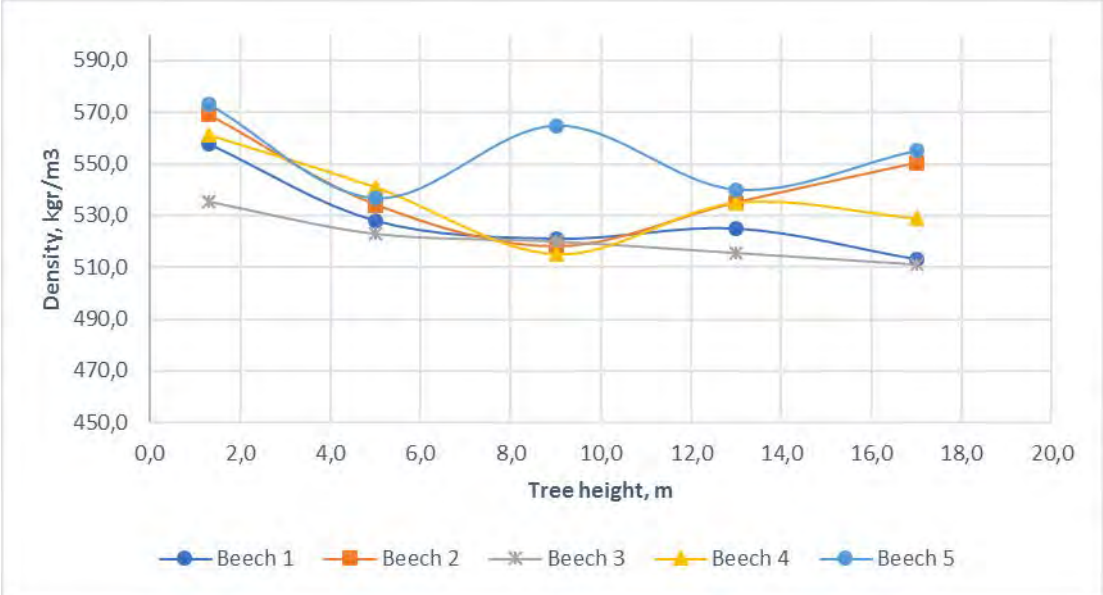

Fig. 7.
Variation of the wood density in beech trees.

The existence of a vertical variation of structural characteristics should be expected, since at different height levels wood is composed of growth rings of different structure. The ring width increases from the top of the tree to a maximum near the lower part of the crown, and then slowly decreases to the base (Tsoumis 1991). Figure 8 shows the results of the ring width variation from base to top of the tree. A vertical variation was observed, as the ring width of beech trees increased from the top of the tree to a maximum of around 9 m, for trees 1, 3 and 4 and then reduced to the base. The same trend was observed in beech tree 5, where the maximum ring width appeared at 5m. For beech tree 2, the ring width declined from the top of the tree, but the highest ring width was found near stem base.

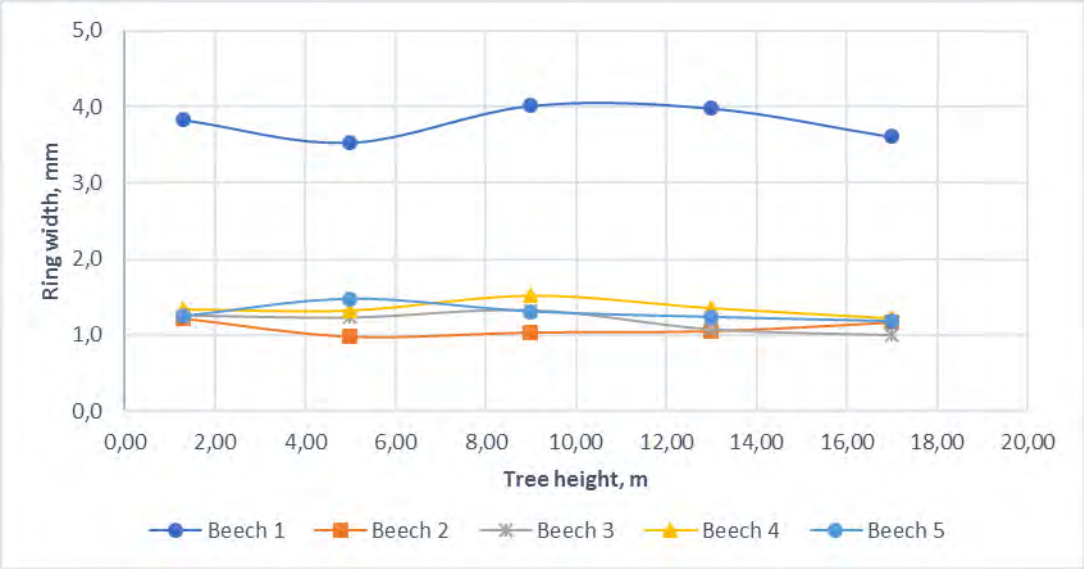

Fig. 8.
Variation of the annual ring width in beech trees.

Variation of the wood density and the annual ring width between beech trees

Based on the results of Figure 7, there was a small variation on wood density between beech trees. For instance, the density values between beech trees 1, 2 and 4 were found quite similar near the base (1,3m) and on 5m, 9m and 13m height from the base. It was also recorded that the wood density for beech tree 3 was lower on every stem height, except from the height of 9m, were the density was quite the same as trees 1, 2 and 4. The wood density of beech tree 5 was the same as other beech trees on the lower parts of the stem (1,3m and 5m). As shown in Figure 8, there were not significant differences on the ring width of different heights of stem discs, between beech trees 2, 3, 4 and 5. While, the ring width of beech tree 1 was found to be the highest on every stem height.

According to Tsoumis (1991), variation of density and ring width between trees of the same species exists and is influenced by environmental conditions (soil, climate, tree spacing) and heredity. This effect of environment is basically expressed through changes of ring width and proportion of latewood and as a result, adjacent tree may differ in pattern of ring width and ring structure.

CONCLUSIONS

The results obtained from this study suggest that the HF densitometry method is a promising technique to assess the density and ring width on stem discs. This method provides the variation of density and analyzes the ring width within trees and in-between trees of the same species. The results of this work indicate that the HF densitometry method allows to measure the density variation on every part of a stem disc, from pith to bark and to differentiate changes in earlywood and latewood density. On the contrary, the gravimetric method calculates the wood density on small parts of the disc or separately for earlywood and latewood. Furthermore, the HF densitometry method is based on a simple measurement procedure, it is extremely fast than the traditional methods and thus it could become widely used for measuring wood density and ring width variations. However, it should be noted that the application of HF densitometry via Lignostation machine to measure wood density variations on stem discs, is highly depended on the milling surface quality and on any abnormalities appeared in wood surfaces of stem discs.

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EFFECTS OF REMOVAL OF DIFFERENT CHEMICAL COMPONENTS ON MOISTURE SORPTION PROPERTY OF *POPULUS EURAMERICANA* CV. UNDER DYNAMIC HYGROTHERMAL CONDITIONS

Tiantian YANG
MOE Key Laboratory of Wooden Material Science and Application
Beijing Forestry University, Beijing, China.100083
E-mail: bjfu100514327@163.com

Erni MA
MOE Key Laboratory of Wooden Material Science and Application,
Beijing Forestry University, Beijing, China.100083
E-mail: *maerni@bjfu.edu.cn*

Abstract

Chemical components have great effects on dynamic wood sorption property. In this study, hemicellulose, lignin and extractive (denoted as DHC, DL and DE, respectively) were removed from Populus euramericana Cv., 20mm in radial (R) and tangential (T) directions with thickness of 4mm along the grain, then was subjected to dynamic cyclic tests where relative humidity (RH) varied from 45% to 75% sinusoidally at 25°C. Based on measured moisture content (MC), the results showed that, various chemical components have different effects on wood sorption behavious. The DL exhibited the largest MC changes as well as the diffusion coefficient, followed by the DE, Control and DHC. Theoretical sorption model was applied and the modeled curves fitted satisfactorily with experimental data. Lignin or extractive removal accelerated the sorption process, while hemicellulose removal caused an opposite result. Dynamic moisture gradient distribution in different treated wood was further investigated with the theoretical model.

Key words: *chemical components removal; dynamic cyclic relative humidity; sorption; conditioning; wood.*

INTRODUCTION

Wood, as one of the environmental friendly natural materials, has been widely used in our daily life for its characteristics of color, pattern, sound absorption, etc., especially the RH conditioning function, provided by that wood is ceaselessly exchanging water with atmosphere, since it follows the ambient relative humidity of the air due to the hygroscopic nature. This in turn brings about the continually changing moisture in wood, and some defects such as cracking and wrapping may be caused, influencing wood using and processing.

Therefore, moisture sorption and wood conditioning function are important information (Kollmann and Cote 1968; Siau 1984), worth studying for the daily application and processing of wood or wood products.

Focusing on this, some studies were conducted and found moisture sorption could be affected by many factors, such as wood own composition, including cellulose, hemicellulose, lignin and extractive (Liu and Zhao 2012). Cellulose and hemicelluloses are rich in free hydroxyl groups, which could make great contribution to the hygroscopicity of wood, whereas lignin is a relative hydrophobic heteropolymer, composed by phenyl propane unit through carbon-carbon bonds or ether bonds. As a result, some studies showed that delignification could weaken the water resistance capabilities of wood. Removing the extractive will encourage wood sorption due to the fact that extractive deposit in cell cavity which could block the passage for water (Ma and Zhao 2012). Studies have shown that heat treatment can effectively reduce hygroscopisity, moisture uptake rate (Bak and Németh 2012), and sorption hysteresis, which could mostly be ascribed to the degradation of hemicellulose. Some researchers also believed that other chemical components of wood such as lignin played an important role in wood hygroscopisity (Repellin and Guyonnet 2005).

Hosseinaei et al. (2012) found that the hygroscopicity of wood-plastic composites decreased as temperature increased because more hemicellulose was removed at higher temperature. Ou et al. (2014) pointed out that the delignification wood-plastic composite had the largest hygroscopicity and swelling, followed by the matrix removal group, extraction group, hemicellulose removal group. In 2014, Zhou et al. (2014) investigated the effect of removing chemical components of lignin and

extractive on EMC of *Cunninghamia lancolata Hook* wood, and indicated the hygroscopicity of delignification wood was larger than the wood with extractive removal.

In addition, wood sorption property could also be affected by the outside RH. In 1988, Skaar indicated that the equilibrium moisture content (EMC) was approximately proportional to ambient RH and pointed out that the RH where the wood exposed was the most significant factor affecting EMC (Skaar 1988). The relation between EMC and RH at constant temperature is generally displayed by a curve named as sorption isotherm (Engelund et al. 2013; Willems 2015). From the sorption isotherm, it is obvious that the EMC of wood increases with increasing RH at a given temperature.

However, the experiments above were conducted under static conditions, where RH and temperature were kept constant, while the atmospheric RH or temperature during wood processing and use is always changing and may be sinusoidal (Schniewind 1967). Therefore, it is quite very necessary to study the wood sorption and effect of chemical components removal under this dynamic condition, which is close to daily use condition of wood.

Chomcharn and Skaar (1983) firstly conducted such work that dynamic sorption and hygroexpansion of wood wafers were studied where RH changed cosinusoidally at a constant temperature. Later in our previous studies, the moisture and tangential (T) and radial (R) dimensional changes of wood subjected to sinusoidal RH variation at a constant temperature (Ma et al. 2010) and square temperature variation at a constant RH (Yang et al. 2015) were investigated. However, these studies concentrated more on studying the wood sorption but little concern had been paid to its relationship with chemical components of wood.

In terms of wood sorption rate, moisture sorption kinetics could be considered as a method. Fick's second law was always been taken as the basement to build mositure sorption kinetics equation, which regarded the diffusion controlled the movement of wood interior, so that the moisture sorption could be simulated accurately. Stamm (1959) explored Fick's diffusion law dealing with the sorption process and illustrated they do not adequatedly describe moisture movement in small wood specimens, other processes in addition to Fickian diffusion operate to limit the rates of moisture change in wood (Nakano 1994a; Zhang et al. 2007). Christensen and Kelsey (1959) studied the rate of water vapour adsorption by small specimens of Klinkii pine in the absence of air and found the sorption rate decreased as the moisture content increased. Kelly and Hart (1969) measured the rate of water vapour adsorption and desorption of yellow poplar and white oak. An empirical equation was derived to fit the experimental data, but only limited success was achieved in relating the constant of the equation to the corresponding relative humidities. In 2010, Ma obtained the moisture sorption kinetics equation by combining the surface sorption theory with Bradley sorption theory. This model suggested that wood adsorption rate was related to the rate at which sorption spaces could be made accessible to water molecules rather than the classical Fickian behavior. And the moisture diffusion coefficient *D* in the model could be affected by species, density, MC, etc. (Ma et al. 2010). Stamm (1959) found *D* increased as MC increased and the exponential relationship could be established at the range from 5-25% MC, and from thence the *D* could also be influenced by wood chemical components for which could make significant contribution to MC.

The study presented here aimed to investigate the effects of removal of different chemical components on wood sorption as well as the conditioning property under dynamic condition. The research results should be helpful in figuring out the effect from different chemical components on wood sorption and enriching the fundamental understanding of the sorptive behavior of wood at non-equilibrium theoretically, and providing improved technical parameters for the conditioning property of wood products in service practically.

MATERIALS AND METHODS

Materials

Poplar (*populus euramericana* Cv.) wood from the Greater Khingan Mountains in China was taken as study species with the average annual ring width of 3.5mm and air-dried density of about 0.4g.cm^{-3}. The specimens were cut from clear, flat-sawed sapwood into the size of 20mm in both R and T directions with thickness of 4mm along the grain.

Chemical treatment of wood

The specimens were divided into four groups according to their pretreatments, including untreated (Control), extractive removed (DE), hemicellulose removed (DHC), and lignin removed (DL). Later the four groups were all boiled in distilled water for 15 min to remove their growth stress.

After this, they were chemically treated as followed:

Extractive removed: the specimens were extracted with a mixture of ethanol and benzene (1:2 volume ratio) (Beijing Lanyi Chemical Products Co., Ltd) for 48h and then boiled for 3h at 60°C in the

same solution heated by water bath to remove soluble extractive. They were then washed with distilled water for 24h, air-dried for 48h, and finally dried at 80°C until a constant weight (m_1) was achieved (Xie 2006).

Hemicellulose removed: the DE was further extracted by liquid hot-water at 170°C in a reactor for 2h with a water-to-solid ratio of 20:1 (w/w) and then washed with distilled water for 24h, air-dried for 48h, and finally dried at 80°C until a constant weight (m_2) was achieved (Hosseinaei et al. 2012).

Lignin removed: the DE was firstly delignified with a mixture of 967ml of distilled water, 20g of $NaClO_2$, and 13ml of CH_3COOH (Beijing Lanyi Chemical Products Co., Ltd) for decompression treatment for 5h and then put into a water bath for 30 h at 40°C and then washed with distilled water for 24h, air-dried for 48h, and dried at 80°C until a constant weight (m_3) was achieved (Zhang et al 2006a, 2006b).

Measurement of dynamic moisture sorption of wood

After chemical treatments, the four groups were conditioned in 45% RH at 25°C controlled by saturated salt solutions of sodium chloride (Macromolecule 1958) purchased from Beijing Lanyi Chemical Products Co., Ltd, respectively over 10 days to obtain equilibrate weights. Afterwards, the specimens were moved into a conditioning oven (DHS 225, YaShiLin Co., Ltd, Beijing) to conduct the cyclic tests as described in Figure 1. The RH (sensitivity ± 1%) and temperature (sensitivity ± 0.5°C) in the oven were programmed to vary in discrete steps according to predetermined schedules, and a thermo-recorder (TR-72Ui, Tandd Co., Ltd, Japan) was placed near the specimens to monitor the RH and temperature. During the processes, weight changes were measured by an electronic analytical balance (ME104E, Mettler-toledo Co., Ltd, America) (sensitivity ± 0.1mg) (Yang et al. 2015). And the measured data could be recorded automatically by personal computer without opening the door of the conditioning oven throughout the dynamic tests. To control the changing frequency of RH, three sinusoidal cyclic periods were designed: 1) for 1h cyclic period, a sinusoidal change completed in 1 hour. One test contains 10 cycles, and last for 10h. Measurements took at every 1 minute. 600 data were collected for each parameter in one test; 2) for 6h cyclic period, a sinusoidal change completed in 6 hours. One test contains 6 cycles, and last for 36h. Measurements took at every 5 minutes. 432 data were collected for each parameter in one test; 3) for 24h cyclic period, a sinusoidal change completed in 24 hours. One test contains 4 cycles, and last for 96h. Measurements took at every 15 minutes. 384 data were collected for each parameter in one test.

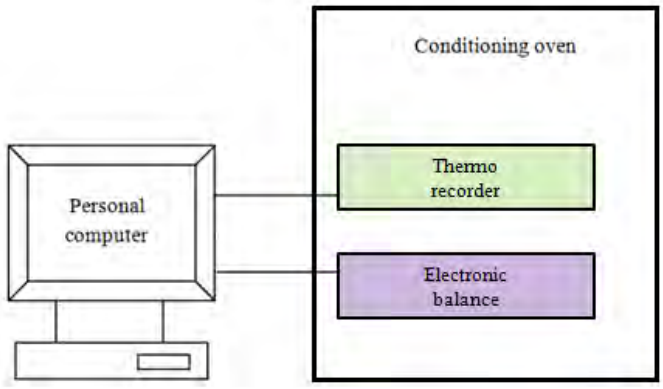

Fig. 1.
Diagram showing the instrumentation for the entire assembly.

In addition, there were three end-matched replicates for each cyclic period. Each test was repeated three times, and average values of the three tests for weights (m_4) of the specimens were taken as the final result.

Finally, the specimens were oven-dried at 103°C for 48h and their oven-dried weights (m_0) were measured for final determination of moisture content.

The ratio of extractive loss (P_1), hemicellulose loss (P_2), and lignin loss (P_3) was calculated according to equations 1 to 3

$$P_1 = \frac{m_0 - m_1}{m_0} \times 100\% \quad (1)$$

$$P_2 = \frac{m_1 - m_2}{m_0} \times 100\% \quad (2)$$

$$P_3 = \frac{m_1 - m_3}{m_0} \times 100\% \quad (3)$$

Approximately 3% of extractive in DE and 9% of the hemicellulose in DHC were removed, each calculated based on the oven-dried weight (m_0) of wood. In DL, lignin losses were 13%.

And the dynamic moisture content of control (MC_1), DE (MC_2), DHC (MC_3), DL (MC_4) was calculated according to equation 4 to 7.

$$MC_1 = \frac{m_4 - m_0}{m_0} \times 100\% \quad (4)$$

$$MC_2 = \frac{m_4 - m_1}{m_1} \times 100\% \quad (5)$$

$$MC_3 = \frac{m_4 - m_2}{m_2} \times 100\% \quad (6)$$

$$MC_4 = \frac{m_4 - m_3}{m_3} \times 100\% \quad (7)$$

Theoretical model for dynamic sorption

The theoretical model for dynamic sorption of Ma et al. (2010) which considered both surface moisture exchange through the air-wood interface and internal diffusion within wood was used in this study to investigate the dynamic moisture sorption. Moisture exchange on the wood surface is given by:

$$m_i = m_{i-1} + a(h - \exp(K_2 K_1^{m_{i-1}} + K_3))\Delta t$$
$$K_1 = 1.0327 - 0.000674T \quad (8)$$
$$K_2 = 17.884 - 0.1432T + 0.0002363T^2$$
$$K_3 = 0.16$$

where: m is MC (%), a is sorption rate constant (1/h), h is RH for the surrounding atmosphere (%), and T is temperature (K). K_1, K_2, and K_3 are given by the Bradley equation (Bramhall 1979), and their values were validated by experimental isotherm curves.

For the transfer of moisture inside the wood, a numerical solution by the finite difference method (Bramhall 1979) for the Fick's second law was used.

$$m_{j_i} = \frac{D(m_{j-1} - 2m_j + m_{j+1})_{i-1}}{\Delta l^2} + (m_j)_{i-1} \quad (9)$$

where: D is the moisture diffusion coefficient (m^2/s), the subscripts i-1, i are ordinal time points, j-1, j, j+1 are ordinal elements in thickness direction, and Δl is the thickness of each element (m). The specimen in this work was divided into 7 ordinal elements along thickness direction as the 1 (surface), 2, 3, and the 4 (center) from surface to center.

The value of D in equation 5 was taken as a constant in the previous study. However, in the present case, it is considered to be MC-dependent and expressed by an exponential function in longitudinal direction (Skaar 1988).

$$D = 0.40 \times 10^{-7} \exp(0.11m) \quad (10)$$

RESULTS AND DISCUSSIONR

General moisture responses

General MC response of poplar wood with different treatments to the sinusoidal RH changes over several cycles is shown in Figure 2. Obviously, MCs of four wood change sinusoidally, but all data shows a phase shift with sinusoidal RH change.

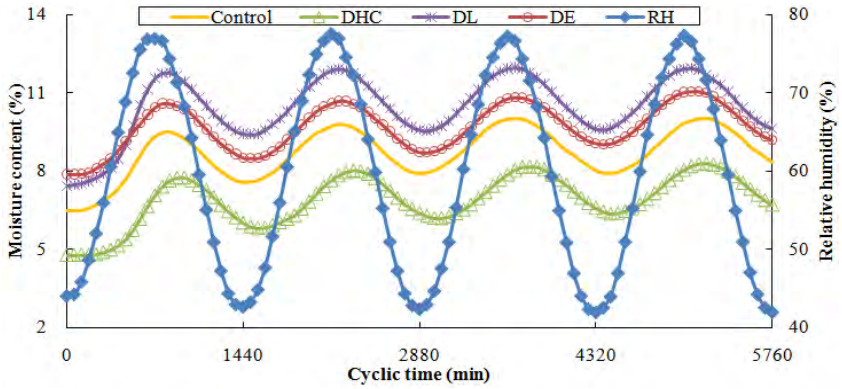

Fig. 2.

Moisture content changes where RH changes sinusoidally at a constant temperature against cyclic time for poplar wood with different treatments (cyclic period of 24 h).

Table 1 summarizes the average MC for the 3 cyclic periods of poplar wood subjected to different treatments. Generally, the measured data shows positive relation to the cyclic period. Expectedly, the DL exhibited the largest MC changes, followed by the DE and then Control. And the MC changes of DHC are the lowest. This is because lignin is a hydrophobic heteropolymer, the delignified group DL was supposed to possess lower water resistance compared with the Control. And extractive would block water passage to certain extent, which can slightly restrain the moisture sorption of wood, while hemicellulose removal can greatly reduce wood hygroscopicity because of a dramatic loss in the quantity of exposed hydroxyl groups.

Table 1

Comparison of average MC with different treatments at 3 cyclic periods

Period (h)	Moisture content (%)			
	Control	DHC	DL	DE
1	8.02 (0.012)	6.24 (0.008)	8.87 (0.019)	8.37 (0.023)
6	8.24 (0.017)	6.32 (0.009)	10.05 (0,021)	9.22 (0.041)
24	8.66 (0.016)	7.04 (0.014)	10.58 (0.018)	9.81 (0.037)

Dynamic theoretical sorption modeling

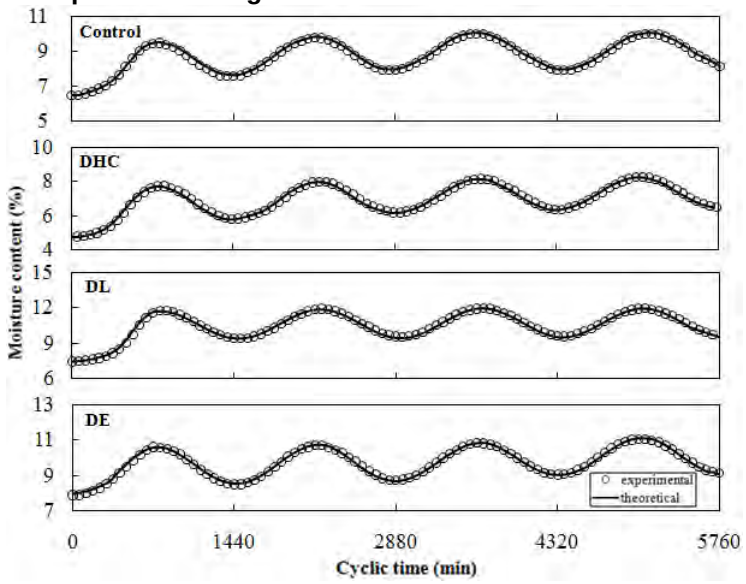

Fig. 5.

Comparison of theoretical curves with experimental results of dynamic sorption for poplar wood with different treatments (cyclic period of 24 h).

The diffusion coefficient *D*, used in dynamic sorption modeling for specimen at three cyclic periods, was also calculated and listed in Table 2.

Table 2

Diffusion coefficient D for poplar wood at 3 cyclic periods under dynamic conditions

Cyclic period (h)	$D \times 10^{-11}$ (m^2/s)			
	Control	DHC	DL	DE
1	0.97 (0.012)	0.79 (0.010)	1.06 (0.024)	1.00 (0.018)
6	0.99 (0.021)	0.80 (0.013)	1.21 (0.033)	1.10 (0.023)
24	1.04 (0.020)	0.87 (0.16)	1.28 (0.034)	1.18 (0.019)
Static value	2.0 (at about 10% MC at 26.7°C) (Christensen and Kelsey 1959)			

It's obvious that the coefficient grows up as cyclic period increases but less than the static value (Ma et al. 2010), and the *D* of DL was the largest, followed by DE, Control, and DHC. This indicates that DL has the highest sorption rate while DHC has the lowest. Moreover, there is a positive relation between *D* and moisture content of specimens when analyzing the Table 1 and Table 2, which agrees with the static results (Stamm 1959). This indicated the effect from chemical components in wood on sorption amount was analogous with sorption rate.

Dynamic moisture gradient distribution in wood

The moisture gradient distributions at four typical time steps (adsorption origin (I), 1/4 adsorption (II), 1/2 adsorption (III), terminal adsorption (IV)) during the adsorption and desorption processes of the second cycle at a period of 24 h, calculated according to equation 8 and 9 (Ma et al. 2010), are shown in Figure 6.

In the case of adsorption taking the Control as an example, the time when the specimens just started to pick up moisture is about 1530 min. At this time, RH has already increased for about 90 min. However, as shown in this figure, moisture still takes on a regular shape of desorption distribution (Jonsson 2004). When adsorption time reaches 1710 min, about 1/4 of the adsorption process, the most relatively uniform distribution is obtained by the adsorption of the surface and desorption of the central part along the thickness direction of the specimens. At 1890 min, the midpoint of adsorption, the entire sample began to sorb water, and a typical adsorption moisture distribution was found. Therefore, initially the central part of the specimens does not respond to the moistening. It takes about 6h until the center part responds. Finally, MC reached the peak value at 2250 min, although RH had already decreased. The moisture gradient at this time is lower than that at 1890 min. Nevertheless, the moisture distribution is still far from equilibrium. However, there are some differences caused by the chemical components removal. For instance, the least MC gradient between surface and central part was existed in DHC (0.70%) and then DL (0.96%), DE (1.04%) followed by control (1.09%). This could be due to the higher MC in central part in second adsorption process resulted from the larger phase lag (section 3.4 for details) for DHC. In addition, the diffusion coefficient was larger for DL ascribed to its lowest lignin content than DE then Control, so that the moisture could reach the central part faster for DL than DE then Control, and the MC difference between surface and center for DL was less than DE then Control. Moreover, it is earlier for DL central part to begin adsorption process (1665min) than DE (1680min) then Control (1695min) and followed by DHC (1710min). Apparently, the maximum time difference existed between DL and DHC was 45min. It could also assume that moisture distributions for desorption have the same tendencies as those for adsorption.

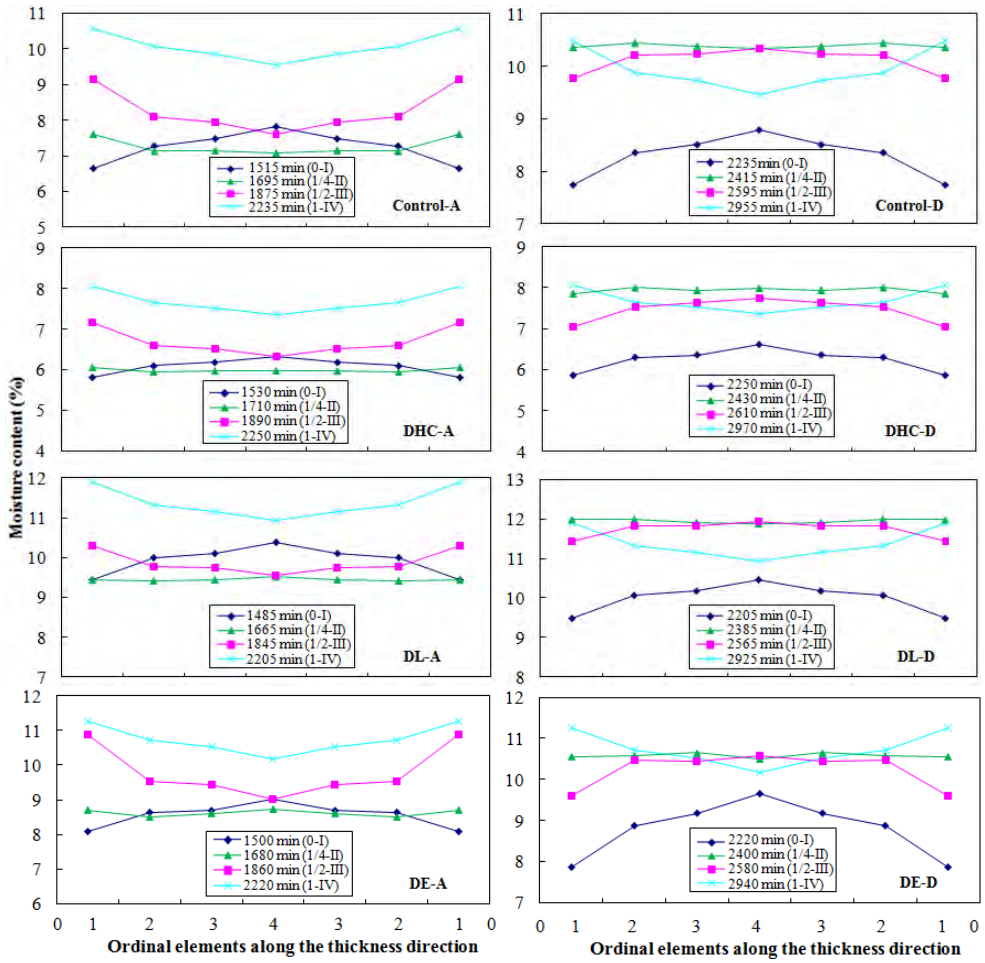

Fig. 6.
Moisture distributions along the thickness direction of the samples treated differently for adsorption (A) and desorption (D) during the second cycle at cyclic period of 24h.

CONCLUSIONS

The effects of removal of different chemical components on dynamic wood sorption where RH varied from 45% to 75% sinusoidally at 25°C were investigated and conditioning property of wood was further discussed. The following conclusions could be attained: MC of both treated and untreated wood also changed sinusoidally, but lagged behind the triggering sinusoidal RH. DL exhibited the largest MC changes (10.58%), followed by DE (9.81%), Control (8.66%) and DHC (7.04%). The theoretical sorption model was applied and the modeled curves agreed satisfactorily with the practice experimental data. It was earliest for DL interior to begin every sorption process, followed by DE, DHC and Control. Diffusion coefficient increased as prolonging cyclic period, and showing the similar variation trend to MC.

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SOME MORPHOLOGICAL PROPERTIES OF PAULOWNIA (*Paulownia elongata* S. Y. Hu) WOOD

Emre BIRINCI
Kastamonu University, Arac Rafet Vergili Vocational High School
Department of Forestry. 37800 Kastamonu, Turkey
Tel: +90 366 362 26 20, Fax: +90 366 362 26 36,
e-mail: ebirinci@kastamonu.edu.tr

Alperen KAYMAKCI
Kastamonu University, Faculty of Forestry
Forest Industry Engineering, Department of Wood Mechanic and Technology
Kastamonu, Turkey
Tel: +90 366 280 17 05, Fax: +90 366 215 23 16,
e-mail: akaymakci@kastamonu.edu.tr

Haci Ismail KESIK
Kastamonu University, Faculty of Forestry
Department of Forest Industry Engineering, 37150 Kastamonu, Turkey
Tel: +90 366 280 17 05, Fax: +90 366 215 23 16,
e-mail: hismailkesik@kastamonu.edu.tr

Ibrahim BEKTAS
Kahramanmaras Sutcu Imam University, Faculty of Forestry
Department of Forest Industry Engineering, 46000 Kahramanmaras, Turkey
Tel: +90 344 280 17 11, Fax: +90 344 280 17 12,
e-mail: ibtas@ksu.edu.tr

Abstract

Pavlonia is a fast-growing tree species cultivated at significant levels in particular forestry in China and Japan. Based on the literature, it is suggested that paulownia wood should be evaluated in furniture production, packaging boxes and boxes, plywood production, chopping, ceiling upholstery and pulp production. Several studies have been conducted to evaluate some fast growing wood species for the paper pulp and paper industry and it has been determined that fiber morphology and chemical composition affect the performance of lignocellulosic materials in pulp and final product quality. For this reason, it is necessary to fully understand the chemical and morphological properties of lignocellulosic materials and their special properties compared to conventional raw materials. In this study, some morphological characteristics of Paulownia elongata wood were investigated.

Key words: *Paulownia elongata; morphological properties; Runkel Ratio; flexibility coefficient.*

INTRODUCTION

The rapid increase of world population, industrialization, unconscious use of forest resources cause rapid depletion of natural resources. This has led to an increase in the openness of wood raw materials in the world. Industrial forestations with fast growing species, which can be subject to private sector forestry, have been developed rapidly in order to eliminate this shortage in the shortest time.

While the supply of wood raw material, which is provided in natural forests throughout the world, is continuously decreasing, the supply of wood raw materials from industrial plantations is increasing. This trend will continue until the entire supply of wood material is dominated in this way (Zobel 1984). Rapidly growing species (willow, poplar, paulownia, eucalyptus, etc.) grown under the world plantation forestry have become an alternative raw material source for the forest products industry (MDF, chipboard, fiber board, paper and cardboard production (Dundar et al. 2016; Kaymakci et al. 2011).

Paulownia is a tree species, otherwise known as the king tree or Chinese imperial tree, and believed to be a gift to China. China and Japan have significant plantations under special forestry (Khan et al. 2003).

Rapid increase in diameter and height is the most important characteristic feature of these species (Minato et al. 2005). These species can easily adapt to places where cultures are made, and at the same time they can provide a very wide distribution (Hua 1981). Today, paulownia is grown in

more than 40 countries and can be planted within the scope of agricultural forestry (Abbasi 2000). Today, around 24 million acres of paulownia are cultivated for various purposes in the world (Kaplan 2008).

Paulownia tree is very adaptable and extremely fast growing hardwood compared to most traditional hardwood and softwood species. When grown in managed plantations, it can be harvested for wood in 4–7 years. By comparison, hardwood trees take 60–80 years to mature (Ayrilmis and Kaymakci 2013).

A lot of work has been done to evaluate some rapidly growing species of wood for paper pulp and paper industry (Jahan et al. 2008). Fiber morphology and chemical composition affect the performance of lignocellulosic materials in pulp and final product quality. For this reason, it is necessary to fully understand the chemical and morphological properties of lignocellulosic materials and their special properties compared to conventional raw materials (Tutus et al. 2004; Khakifirooz et al. 2012).

The mechanical strength of the dough and paper is a major factor in the natural material fiber size, runkel ratio and elasticity coefficient (Caparrosu et al. 2008). Dimensional measurements of the fibers help assess various properties of the paper. The most important observation made to find the suitability of any raw material in the production of paper clay is the runkel ratio (calculated by the ratio of lumen width to twice the cell wall thickness). The standard value of this ratio is 1. Appropriate dough strength properties are generally obtained when the runkel ratio is below the standard value.

In general, high runkel ratio fibers are stiffer, less flexible, and form bulkier paper of lower bonded area than low runkel ratio fibers. This effect is related to the degree of fiber collapse during paper drying, a phenomenon affected by the cell wall thickness and degree of refining that fibers undergo prior to papermaking (Binotto and Nicholls 1977). Materials having runkel ratio less than 1 would be suitable for papermaking, because they collapse (become ribbon like) and provide a large surface area for bonding (Jang and Seth 1998). Macerated paulownia fibers have a runkel ratio of 0.34. Sheets made from short and thin-walled fibers of paulownia may be expected to give relatively dense papers which are weak in tearing strength, but are superior in burst and tensile properties. From this point of view, the fibers are suitable for papermaking (Ashori and Nourbakhsh 2009).

Flexibility coefficient is the percentage of lumen width over fiber width. It expresses the potential of fiber to collapse during beating, or during drying of the paper web. Collapsed fibers provide more bonding area and subsequently stronger papers are produced. On the other hand, strength properties of paper such as tensile strength, bursting strength and folding endurance are affected mainly by the way in which individual fibers are bonded together in paper sheets. The degree of fiber bonding depends largely on the flexibility of individual fibers. The coefficient of flexibility is 75 and therefore it is expected to yield well-bonded fibers and form a smooth printing surface. It is worth noting that the average of this value for hardwoods and softwoods is 55–70 and 75, respectively (Ashori and Nourbakhsh 2009).

OBJECTIVE

The purpose of this study is to investigate some morphological features of *Paulownia elongata* wood.

MATERIAL, METHOD, EQUIPMENT

In this study, a total of 3 species of *Paulownia elongata* were provided from Izmir, Turkey. 2-4m sections from experimental trees were used to determine morphological characteristics. Fiber morphology is one of the properties that must be known to determine the use of wood in cellulose, paper and cardboard industries. Knowing the dimensions of cellulosic fibers gives important clues in terms of the quality and quantity of the paper to be obtained. For this reason, four groups of fiber properties were determined in the wood samples of *Paulownia elongata* as fiber length, fiber width, lumen width and cell wall thickness. As a result of keeping the samples in the hot water bath (80 ° C), sodium chloride ($NaClO_2$) affects the wood structure lignin and the cellulose fibers become free. Permanent preparations were also prepared with the help of adhesives for each fiber obtained in this way. The fiber size, fiber width, lumen width and cell wall thickness of the prepared preparations were measured by means of a microscope with micrometric oxides. 30 measurements were made in each trial tree. Using the data obtained from the measurements, the flexibility coefficient and slenderness ratio of the fibers, rigidity and runkel ratio were determined.

RESULTS AND DISCUSSION

Indings were obtained by performing measurements on 30 robust fibers for each experimental group. The morphological characteristics of the *Paulownia elongata* samples are shown in Table 1.

Table 1

Morphological characteristics of Paulownia elongata wood from Izmir

Code		Cell Wall Thickness (μm)	Lumen Width (μm)	Fiber Width (μm)	Fiber Length (mm)
Paulownia elongata (from Izmir)	**Average**	4.85	36.78	46.33	1.04
	Standad Deviation	1.36	6.61	7.39	0.17

When Table 1 was examined, cell wall thickness, lumen width, fiber width and fiber length of pavalion wood were determined as 4.85μm, 36.78μm, 46.33μm and 1.04mm respectively.

Table 2

For some tree species and annual plants the fiber length specified is the fiber width, lumen diameter and cell wall thickness values

Species	Cell Wall Thickness (μm)	Lumen Width (μm)	Fiber Width (μm)	Fiber Length (mm)	References
P.elongata	4.85	36.78	46.33	1.04	**This study**
P.elongata	6.31	33.00	45.68	1.06	**Kaymakci 2010**
P.elongata	4.81	32.00	41.09	1,00	**Kaymakci 2010**
P.elongata	**2.75**	**30.00**	**35.00**	**1.03**	**Nasir and Mahmood**
P. fortunei	**3.69**	**26.00**	**33.00**	**1.04**	Nasir and Mahmood 2000
P.tomentosa	**4.05**	**31.00**	**39.00**	**0.95**	Nasir and Mahmood 2000
E. globulus	**-**	**7.00**	**18.00**	**1.28**	**Teresa et al. 2000**
Canola stalk	**5.26**	**12.50**	**23.02**	**1.17**	**Enayati et al. 2009**
Corn stalk	**1.32**	**10.70**	**24.30**	**1.32**	**Usta et al. 1990**
Kenaf	**2.60**	**20.00**	**-**	**-**	**Athicson 1993**
Softwood	**13 - 17**	**15-30**	**32-43**	**3-7**	**As et al. 2002**
Hardwood	**-**	**-**	**20-40**	**0.7–**	**Athicson 1987**

Table-2 was prepared to compare the morphological characteristics of Paulownia elongata species with some species. It is thought that the differences between cell wall thickness, lumen width, fiber thickness and fiber length values may be due to the age of the tree, growing environment conditions. Bozlar et al. (2014), the effects of the growing environment on the anatomical characteristics of wood in the alder plantations were determined. Changes in anatomical characteristics are related to latitude, longitude grades, elevation, and macro climate types. It has been determined that the local anatomical characteristics of the wood are influenced by factors such as height, precipitation, temperature, viewing, as well as local environmental conditions (Bozlar et al. 2014).

The growth process in the trees has a very complex structure with the effect of numerous internal and external factors. The suitability and quality of the wood material for a particular purpose is also influenced by the variation of one or more of these factors, which can lead to differences in many properties of the wood. Tree age, genetic characteristics and environmental conditions can cause changes in wood properties. At the same time, the effects of factors such as fire, drought, frost, and damage caused by forest animals are also different from each other in the structure of wood. There are a number of criteria that determine the suitability of wood for a given place of use. These criteria include density, uniformity of annual rings, heartwood ratio, fiber length, presence of young wood and reaction wood, knottyness, fiber curvature, chemical composition and quality of extractive substances. It is possible to control these factors with various measures. (Dogu 2002).

The measured values for the morphological properties of the fibers of the paulownia wood are shown in Table 3.

Table 3

Measured values of morphological properties of Paulownia elongata wood

Growth Place	Tree Age	Slenderness Ratio	Flexibility Coefficient	Runkel Rate	Rigidity
Paulownia elongata (from Izmir)	11	22.71	79.58	0.27	10.65

As is known, those having an elasticity ratio greater than 75 are very flexible fibers, 50-75 are elastic fibers, 30-50 are rigid fibers, and those less than 30 are defined as very rigid fibers (Kırcı 2000). According to this definition, it can be said that pavlion wood generally enters into very flexible fibers (76.13).

While Slenderness is over 100 for softwood, hardwood is very rarely higher than 70. The higher the felting ratio, the higher the strength of the paper, the higher the tearing, breaking and double folding resistance (Gencer 2010). When the Paulownia wood slenderness ratio (24.17) is examined, it is estimated that the resistance properties of the paper produced will be low.

The runkel value of the paulownia wood fibers was found to be 0.33 in general. When the runkel value is lower than 1, it is known that the fibers are thin-walled and very suitable for paper-making. In this way, it can be said that paulownia wood is suitable for paper-making.

CONCLUSIONS

The constant increase in demand for wood raw materials in the world creates a continuous supply shortage that is ripe for supply imbalance.

Among the measures taken to shut down this openness resulting from supply and demand imbalance is undoubtedly to do industrial afforestation with fast developing species. The greatest environmental benefit of plantation forestry is the ability to replace rapidly growing species with economically valuable tree species. In this way, the pressure on the forests has been reduced and at the same time a social responsibility awareness has been fulfilled. When examined in this respect, paulownia emerges as a species that can substitute for the higher economic value species. Paulownia can be used successfully in all the branches in the forest products business area, especially in the furniture sector. However, the wood properties should be carefully considered when selecting the activity area. In this study, the morphological characteristics of pavlion wood were investigated. Morphological features are crucial for determining the potential of wood to be used in the paper industry. The information obtained as a result of the tests showed that Paulownia elongata wood is an alternative raw material for paper production. However, when angiospermae wood fibers are thought to be composed of short fibers, it may be advisable to use gymnospermae in combination with tree species in order to overcome this disadvantage. By specifying other properties of this species, many different uses and advantages can be determined.

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WOODWORM (*Anobium punctatum*) INFESTATIONS IN CENTRALLY-HEATED AND UNHEATED PROPERTIES IN THE UK

Paul CARTER
formerly **Paul Carter Associates**
Tel 07875 206875, E-mail: pc@paulcarterassociates.co.uk.

Andy PITMAN
Fibre 7 UK Ltd, Atlantic Way, Barry CF63 3RF
Tel 07951 701992, E-mail: andy.pitman@fibre7.com

Gervais SAWYER
Tel 07751 670025 E-mail: gervais.sawyer@blueyonder.co.uk

Abstract

The influence of central heating on the incidence of active woodworm attack in 204 UK houses built between 1750 and 1960 was examined through an inspection of ground floor and roof space timbers. The work was undertaken in response to reports in the UK media that central heating makes building timbers unsuitable for colonisation and development of woodworm in UK houses. The study found active infestation in roof timbers and/or floor timbers in 77% of the centrally-heated houses surveyed. This clearly shows that timbers in these parts of houses remain susceptible despite these being centrally heated

Key words: *anobium punctatum (De Geer); woodworm; common furniture beetle; timber-survey.*

INTRODUCTION

Woodworm *Anobium punctatum* De Geer which is also referred to as common furniture beetle is a pest of hardwood and softwood building timbers and is widespread in distribution (Eaton and Hale, 1993). The importance of this beetle as a UK timber pest is reflected in two publications assigning large sections to its pest status in the UK being published (Hickin 1963, Hickin 1967).

Several UK media reports (e.g. BBC 2's *Raising the Roof* documentary; Hutton 2008; Howell 2014) have suggested the use of central heating in UK buildings has led to localised climatic conditions unfavourable for the colonisation/survival of *A. punctatum* in these buildings. It was further suggested that central heating has meant the incidence of active woodworm infestations in UK houses is now increasingly rare.

The reason cited for this, is that the moisture content of timbers is lowered in centrally-heated buildings due to the increase in air temperature, thereby making it less favourable for colonisation and development of immature stages of *Anobium.*

However, risk of attack by woodworm will also depend on other factors, some of which may also be linked to installation of central heating and which includes:-

1. mated females having access to the timbers to initiate attack through laying of eggs, which is likely to be influenced by location in the building.
2. Whether infested wood items are brought into buildings to act as a reservoir (source) for infestation. It should be noted that firewood brought into buildings and left in fireplaces over summer months and birds nest material in chimneys have all been reported as reservoirs for wood boring insects (pers comm David Pinniger).
3. Whether wood/wood product is suitable to support the development of the beetles which will depend on whether it is susceptible as a species, has been over-coated, received protection with preservatives containing insecticides as a pre-treatment or remedial treatment, whether it is of an appropriate moisture content to support development of woodworm.

With respect to the development of woodworm, several studies have examined the influence of environmental conditions on the development of various stages of the life-cycle (egg, larva, pupa and adult) and life-cycle completion i.e. egg through to adult.

These studies have shown that rate of development is greater at higher temperatures and that low humidity promotes greater mortality in some stages of the life cycle e.g. eggs (Hickin 1963).

Studies have also found that the moisture content of the timber influences the rate of development i.e. the time to complete the life cycle. Rate of life-cycle completion is greater where the

wood moisture content is elevated resulting in emergence of a greater number of egg laying females available to re-infest timber over time. Life cycle completion normally takes longer where wood is drier. However, *Anobium* is adapted to develop in wood of low moisture content. The European test standards (EN 49-1: 1985) to evaluate preservative efficacy against this species requires rearing of immature stages in European oak sapwood at 21±2°C and 80±5 relative humidity.

Wood is hygroscopic and its moisture content will be influenced by environmental conditions (temperature and relative humidity), wood species and age of the timber.

Studies have examined the environmental conditions and corresponding effects on wood moisture content in different regions of buildings and for different building types. In roof spaces temperatures have been found to vary with time of year and roof covering and were reported to have an influence on susceptibility of timbers to other wood boring beetle pests such as *Hylotrupes bajulus.*

OBJECTIVES

Detailed surveys of different building types for *A. punctatum* activity were conducted including those of different ages, with and without central heating in order to:-

i.) establish whether the hypotheses put forward in the UK-media that central heating makes buildings unsuitable for colonisation and attack by *A. punctatum* is correct.

ii.) establish whether central heating could be employed as an active medium for the eradication of woodworm infestations in houses.

METHOD, MATERIALS AND EQUIPMENT

Surveys of UK properties built between 1750-1962 were conducted in 2001, 2002, 2003 and 2005. Properties where central heating had been installed were recorded. Inspections were conducted of ground floor timbers and those in roof spaces.

Active Common furniture beetle attack was detected by examining timbers and underlying surfaces for the presence of new emergence holes and freshly produced frass (wood pellets - excreta and wood flour ejected from holes). Hole shape and diameter was used to confirm that these resulted from emergence of adults of this species.

In 2001, 2002 and 2003, each property was the subject of examination of roof and ground floor timbers. It should be noted that not all ground floor timbers were available for monitoring because of the presence of floor coverings. In 2005 only roof voids were investigated.

Where properties had central heating systems installed, central heating had been in place for over 20 years and the effects of such heating were therefore well established. Surveys were carried out during periods of the year when these heating systems would have been in full use.

The relative humidities and temperatures in the roof voids were taken at time of inspection along with the moisture content of the timbers using calibrated meters.

RESULTS AND DISCUSSION

Table 1

Survey data collected between January – March 2001

Age of Property	No. of houses surveyed	Centrally heated with active A. punctatum	Centrally heated with no or old A. punctatum activity	No central heating with active A. punctatum	No central heating with no or old A. punctatum activity	% Moisture content Roof timbers Min/max	% Moisture content ground floor timbers Min/max	Average relative humidity (%) in roofspace	Average roof temp °C
1800-1849	9	3	3	0	3	13/21.1	14.2/19	71	11.8
1850-1900	16	7	3	4	2	18/23.3	13.4/15.5	79	15
1901-1940	16	8	1	7	0	9.6/21	13.9/15.8	79	14
1950-1962	5	5	0	0	0	12.2/15.2	-	74	12
Totals	46	23	7	11	5				
Percentage of those surveyed		50%	15%	24%	11%				

Table 2

Survey data collected between January-April 2002

Age of Property	No. of Houses	Centrally heated with active A. punctatum	Centrally heated with no or old A. punctatum activity	No central heating with active A. punctatum	No central heating with no or old A. punctatum activity	Moisture content Roof timbers Min/max %	Moisture content Ground floor timbers Min/max %	Average relative humidity (%) in roof space	Average roof temp° C
1750-1900	28	12	5	4	7	11 / 21.1	N/A	68	11.5
1901-1940	17	14	1	2	0	10 / 21.0	13.9 / 19.2	77	13.5
1950-1968	15	13	2	0	0	11.3 / 15.2	10.3 / 14.8	79	12.6
Totals	60	39	8	6	7				
Percentage of those surveyed		85%	17%	13%	15%				

Table 3

Survey data collected between February-May 2003

Age of Property	No. of Houses	Centrally heated with active A. punctatum	Centrally heated with no or old A. punctatum activity	No central heating with active A. punctatum	No central heating with no or old A. punctatum activity	Moisture content Roof timbers Min/max %	Moisture content Ground floor timbers Min/max %	Average relative humidity (%) in roof space	Average roof temp° C
1800 -1900	33	15	8	3	7	10 / 21.1	N/A	70	11.6
1901-1950	14	9	2	2	1	11 /21.0	12.2 / 22.2	75	12.9
1951-1962	6	6	0	0	0	9.8 / 15.2	N/A	78	12.5
Totals	53	30	10	5	8				
Percentage of those surveyed		65%	22%	11%	17%				

Table 4

Survey data (roof space only) collected between January- April 2005 (roof surveys only)

Age of Property	No. of Houses	Centrally heated with active A. punctatum	Centrally heated with no or old A. punctatum activity	No central heating with active A. punctatum	No central heating with no or inactive A. punctatum	% Moisture content of Roof timbers Min/max	Moisture content Ground floor timbers Min//max	Average relative humidity (%) in roof space	Average roof temp °C)
1800 -1900	15	5	4	3	3	11 -22	N/A	75	11.8
1901-1950	21	13	6	1	1	11 – 21	N/A	77	12
1951-1970	9	8	0	1	0	10 - 21	N/A	80	12.4
Totals	45	26	10	5	4				
Percentage of those surveyed		57%	22%	11%	9%				

DISCUSSION

Most of properties inspected were during pre-purchase surveys. In the UK, the bank loaning money requires the house buyer to supply an independent survey to establish condition of the property. Of 204 houses inspected, 145 showed evidence of active infestation (71%) in either floor or roof timbers. Of these 118 out of 153 centrally heated houses (77%) were affected, while 27 out of 51 without central heating (53%) were affected. This shows that *A. punctatum* can complete its life-cycle in roof space timbers and ground floor timbers in centrally heated properties.

Timber is hygroscopic meaning moisture content changes with changing environmental conditions. Wood moisture content is at equilibrium with surroundings. However, in regions of the building where environmental conditions change during the year, then so will equilibrium moisture content. In the UK roof spaces and sub-floor void spaces are designed to be ventilated to promote ventilation drying in case of any leaks. This means the environmental conditions will closely match those outside of the building and timbers here will be exposed to different conditions to those in the heated region of the building. In the substantial majority, roof and floor timbers had moisture contents more than 12% despite the influence of central heating.

Ventilation through eaves and air-bricks to allow for air flow also provides access to flying adults able to lay eggs and infest timbers. In addition, these timbers are rough-sawn and uncoated facilitating egg laying and infestation.

CONCLUSIONS

1. There is no evidence that the use of central heating creates conditions unsuitable for the survival of woodworm in houses.
2. Central heating cannot be employed as an active medium for the eradication of woodworm in houses.
3. The incidence of woodworm remains widespread.

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SECTION 2. WOOD DRYING AND HEAT TREATMENTS

EVIDENCE OF CLIMATIC EFFECT ON VISCOELASTIC ORTHOTROPIC MATERIALS: NUMERICAL INVESTIGATION OF THERMO-HYDRO LOADINGS ON EUROPEAN WOOD SPECIES

Seif Eddine HAMDI
Université Clermont Auvergne, CNRS, Institut Pascal, F-63000 Clermont-Ferrand, France
E-mail: seif3eddine@yahoo.com

Rostand MOUTOU PITTI
Université Clermont Auvergne, CNRS, Institut Pascal, F-63000 Clermont-Ferrand, France
E-mail: rostand.moutou_pitti@uca.fr
CENAREST, IRT, BP 14070, Libreville, Gabon

Malick DIAKHATE
Université Bretagne Occidentale, FRE CNRS 3744, IRDL, F-29600 Morlaix, France
E-mail: malick.diakhate@univ-brest.fr

Manja KITEK KUZMAN
University of Ljubljana, BF, Department of WST, Slovenia
E-mail: Manja.Kuzman@bf.uni-lj.si

Abstract

The mixed mode configurations coupling mechanical, hydric and thermal effects for viscoelastic orthotropic materials like wood is studied. The numerical application are proposed on european species (Abbie Miles alba). The analytical formulation of the energy release rate is introduced by A integrals generalized to mixed mode crack growth. The viscoelastic effects are introduced according to the generalized Kelvin Voigt model. This new formulation is based on conservation laws and real and virtual mechanical and thermal fields. The Mixed Mode Crack Growth specimen, providing the decrease of energy release rate during crack propagation, is considered in order to compute the various mixed mode ratios. The analytical formulation is implemented in finite element software Cast3m and the crack growth is obtained by testing the Griffith criterion rewritten in time domain under orthotropic configuration. As results, the evolution of energy release rate and the stress intensity factors versus crack length and hydric variations are computed for the proposed european wood species.

Key words: *mixed mode fracture; viscoelasticity; environnemental effects; european species.*

INTRODUCTION

Currently, worldwide, and arguably European industries, are showing increasing interest in wood based structures. Economic and environmental contexts have enabled the emergence of new markets for green constructions that have thus far been confined for steel and concrete based structures. The work on improving the mechanical properties of a tropical wood species, such as Okume (Aucoumea klaineana), Iroko (Milicia excelsa) and Padouk (Pterocarpus soyauxii) arguably offers many advantages, including lower cost and environmental impact (Nziengui et al. 2017). The benefits may also include energy savings, renewability of the resource, reducing the content of raw fossil materials and recycling (Odounga et al. 2017). However, wood materials also present drawbacks, such thermal and hydric sensitivity and multi-feature heterogeneity, compared with conventional civil engineering structures as steel and concrete. Fundamentally, the full potential of wood-based materials has still not been completely exploited because the relationships between fracture parameters at the microscale and macroscale behavior remain poorly described or integrated.

For civil engineering construction, wood is a traditional material that has been widely used for various types of structures. For example, there are about 27,000 timber bridges over a total of 40,000 bridges in Australia (Ranjith et al. 2011). By observing historical buildings around the world, it can be seen that wood-based structures guarantee a long-term service life with a high durability level. However, in timber structures, cracking initiation and propagation are frequently recognized as main causes leading to structural failure (Riahi et al. 2016). Mechanical behaviors of cracked timber structures are affected by environmental conditions: moisture, temperature, etc. These hydrothermal

conditions could produce a higher risk of crack growth in timber structures (Dubois et al. 2009). The fracture studies in wood structure therefore should take into account these environmental conditions.

Wood is considered as an orthotropic hydro-mechanical material whose mechanical behavior strongly depends on the moisture content and the temperature. Taking into account humidity and temperature variation, the mechanical behavior assessment becomes more complex due to the coupling effect between the mechanical stress and the hydric state (thermos-hydro-mechanical behavior (THM)) (Moutou Pitti et al. 2009; Hamdi et al. 2009). The viscoelastic behavior of wood under variable humidity, known as the mechano-sorption behavior, induces different responses in the drying and in the humidification phase. However, in presence of climatic variations, the long terms load and especially the crack initiations, the mechanical behavior of wooden structures is found highly modified, disturbing their implementation and shortening their life in service. The effects of moisture changes on the propagation of cracks are not yet clearly identified. Therefore, it appears necessary to investigate the influence of the variable environment and crack growth process on the mechanical properties of wood structures.

Subsequent research has been performed by employing finite element calculations to determine the fracture mechanics parameters for a variety of wood species and for different grain orientations. Moutou Pitti et al. (2007a,b), showed that at least some of the assumptions underlying the use of the linear elastic fracture mechanics for wood, namely, the assumption of plane strain, and orthotropic and linear viscoelastic behavior, may be considered valid for wood characterization. However, very few studies were found in the literature on the development and the analysis of cracking process during the variation of temperatures and moisture in wood-based materials (Tjeerdsma et al. (1998); Triboulot et al. (1984); Popovic et al. (2006)). In this paper, a numerical analysis of a thermal expansion effect on crack growth in a wooden plate is discussed by using energetic approach. In the first section, the mathematical formulation of the invariant integrals, based on a conservative law approach, is introduced. A combination of real and auxiliary displacement fields (Noether (1971)), is employed in order to isolate different fracture modes by introducing a generalization of the virtual work principle. The Noether's theorem is used to define the general form of the A-integral. The generalization for a viscoelastic behavior is proposed in the second section. The numerical solution is based on a viscoelastic crack growth algorithm which computes step by step the viscoelastic response, the separation of the energy release rate and the crack growth process by remeshing. According to finite element method, the time domain resolution is given by the proposed algorithm. Finally, a numerical validation, based on a MMCG specimen under temperature and moisture variations, is presented. Results are presented in terms of a parametric analysis of the energy release rate evolutions versus time, during the crack tip advance, for different mixed-mode configurations in european and tropical wood samples.

MATERIAL AND METHOD

Numerical analysis and A-integral formulation

The formulation of the A-integral is based on the analytical work developed by Moutou Pitti et al. (2010), for mechanical and thermal loadings effects estimation. Considering a two-dimensional cracked volume $\boldsymbol{\Omega}$ with a rectilinear crack of length a, subjected to external loads $\boldsymbol{\sigma}^{\infty}$ and $\boldsymbol{\gamma}^{\infty}$, applied far from the crack, and Γ a path which surrounds the crack tip oriented by the normal $\vec{n}$ of component $n_j, (\mathrm{j} = 1,2)$. With these considerations, according to Lagrangian Euleurian hypothesis (Donea et al. (1982)) and conservative laws (Noether (1971); Bui (2007)), in stationary crack, the A-integral is given by Moutou Pitti et al. (2010):

$$A = \int_{\Gamma} \frac{1}{2}\left[\sigma^{v}_{ij,k}\, u_i - \sigma^{u}_{ij}\, v_{i,k}\right]\theta_{k,j}\, dS - \int_{\Gamma} \frac{1}{2}\left[\gamma\vartheta_i \delta_{ij} v_{i,jk} \Delta T_{,j}\right]\theta_{k,j}\, dS + \int_{L} \frac{1}{2} F_i\, v_{i,j}\, \theta_j\, dx_1$$

where σ^{u}_{ij} and σ^{ϑ}_{ij} are stress tensor components deduced from the real displacement field u and the virtual displacement field ϑ, respectively. $F_1 = p$ and $F_2 = q$ on the upper lip and $F_1 = -p$ and $F_2 = -q$ on the lower lip. The first term of the A-integral is the classical term of the Mθ-integral which facilitate the separation of the contribution of each fracture mode, without resorting to separate the displacement field into a symmetric and antisymmetric parts. The second term of the A-integral deals with the temperature effect, including temperature gradients inducing thermal dilatation and contraction. The last term of the A-integral represents the effect of pressures p and q applied perpendicularly to the cracked lips. Note that, the mechanical load applied on the cracked lips can be

induced by fluid action or contact between the crack lips during the crack growth process. The only restriction is the non-existence of friction or shear effects in the cracked lips.

Mixed Mode Crack Growth specimen

The MMCG specimen of tropical wood sample presented in Fig 1a, is a combination of wood CTS specimen developed by DCB specimen Moutou Pitti et al. (2010), is used in order to obtain different mixed mode ratios and crack growth stability. The MMCG design stability is obtained by proposing a variable section. However, the geometry must concentrate the stress singularity around the crack tip in order to obtain an initial instability by using the arcan device, as depicted in Fig 1b. The numerical analysis is performed under plane stress conditions and based on the finite element mesh depicted in Fig 2a. For the numerical simulations, the A-integral method is implemented in the finite element software Cast3m (Cast3m (2010)). The external load is a creep loading applied to a perfect rigid arm with a chosen initial crack length of 40 mm is chosen. Points A_α and B_α with α = (1...7) are holes where forces can be applied with the angle β oriented according to the trigonometrically direction for different mixed-mode ratios. The pure opening mode is obtained by applying opposite forces in $A_1$and $B_1$with $\beta = 0^\circ$, as shown in Fig 2c. In the same way, loading points A_7 and B_7, with loading angle $\beta = 90^\circ$, are employed in order to impose a pure shear mode, as depicted in Fig 2c. Intermediate positions induce different mixed mode ratios.

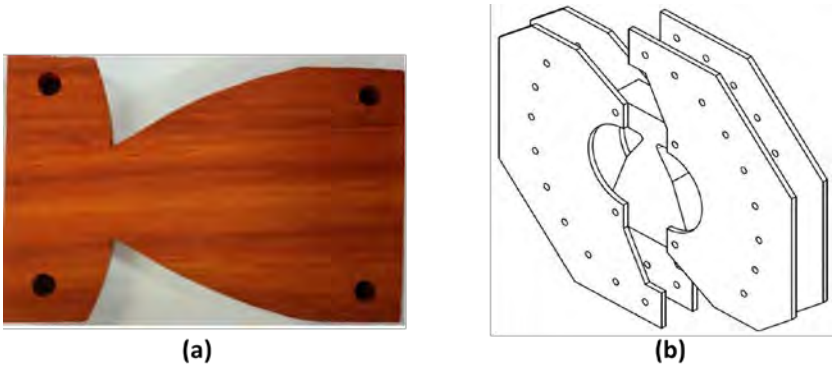

Fig. 1.
(a) MMCG wood specimen – (b) Modified Arcan fixture.

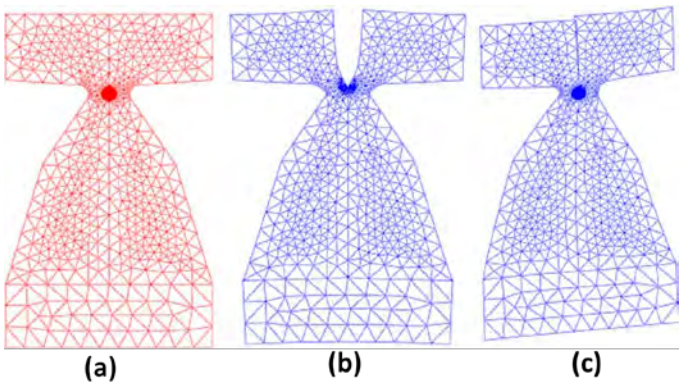

Fig. 2.
(a) Finite elements mesh of the MMCG specimen – (b) Virtual displacements for opening mode – (c) Virtual displacements for shear mode.

RESULTS AND DISCUSSION

Crack growth analysis under thermal loads

Considering physical phenomena inducing wood structures expands as the temperature rises, and restraint against contraction on subsequent cooling, cracks formation and development are initiated by various crack driving forces. In fact, when the crack became stable and had to be induced to propagate beyond it, this implied that the stable crack tip had been arrested at a zone of high toughness; in order to propagate further, the crack preferred the path of the least resistance, which

bypasses the tough zone. Then, it is necessary to quantify the crack driving mechanisms such as temperature level and crack growth speed.

The simulation of a temperature dilatation effect on the energy release rate versus the crack growth speed and crack tip advance during mechanical loadings. the analysis deals with crack growth behavior during the heating process. Fig 3a and Fig 3b show the variation of energy release rate versus time and different crack tip advance for mixed mode during the heating process ($\Delta T = +20^{\circ}C$) under creep loadings. The initial crack length is fixed to 40 mm. Successive cracks increments of $\Delta a = 1\ mm$ and $\Delta a = 8\ mm$ have been chosen and conducted up to a final crack length of $112\ mm$. In order to examine the uncoupling process, the simulation is limited to a mixed-mode configuration $(\beta = 45^{\circ})$. According to past simulations, the loading is constant with unitary opposing forces. For next results, the integration domain is based on crown C_6. The indicated time designates the corresponding simulations are proposed for different constant crack tip speed characterized by the imposed time increment step as shown in Fig 11. The results of part of the open mode presented in Fig 3a in terms of energy release rate versus time increment, shows that under higher temperature expansion a slow crack step advance leads to a higher crack driving force. Moreover, part of the shear mode presented in Fig 3b, shows a relative stability of energy release rate evolution over time, which illustrate the effect of a thermal expansion process in a progressive incohesion of cracked lips around the crack process zone.

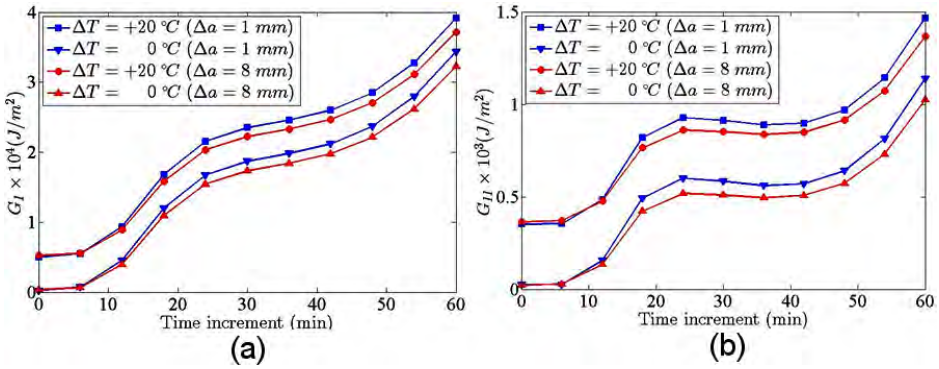

Fig. 3.
Energy release rate versus time and different crack tip advance (Δa =1 mm and Δa =8 mm), for mixed mode during heating process (ΔT =+20°C): Open mode part (a), Shear Mode part (b).

Crack growth analysis under hydrics loads

The analysis of cracks propagation of in mixed mode coupling the mechanical and moisture loads in the wood via the MMCG sample is carried out using an incremental finite element approach using the A- integral. This fact simultaneously leads to the possibility of separating the rupture process and the viscoelastic effect. The hydric fields calculated in the elastic phase before crack propagation are projected on the MMCG mesh in order to calculate the cohesion stress which incorporates this time a selected humidity variation. It should be noted that the viscoelastic procedure is applied before the next moisture step is taken into account and the cracking parameters in terms of viscoelastic energy release rate (G) are evaluated at each step until the test piece is ruined. The effect of Thermo-Visco-Hydro-Mechanical Load Coupling is observed in the wood material for all mixed mode configurations. In this case, Fig. 4a and 4b show the evolution of energy release rates in opening mode (GI), and shear mode (GII) as a function of crack length, for different moisture levels and different mixing rates using the invariant integral A. We note initially, a gradual growth of the development zone (growth phase of energy release rates) and, in a second phase, a stationary phase with a stabilizing changing release rate 'energy. More precisely, we observe, a higher rate of energy restitution for the mode II part (G_II), indicating that the cracking phenomenon is driven by this mode. It's to be noted that G increases in proportion to the moisture content with a higher proportion for ΔT = 30 °C.

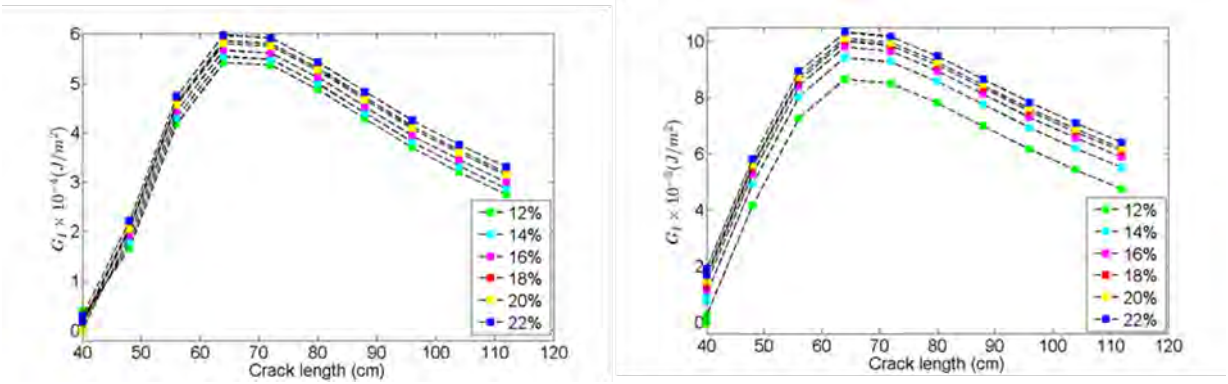

Fig. 4.
Effect of variation in moisture content (MC) on energy recovery rate (G) versus crack length: ΔT = 10 °C (a), ΔT = 30 °C (b).

CONCLUSIONS

This work attempted to numerically investigate the influence of temperature and moisture on crack driving forces of wood-based materials. An analytical formulation of the A-integral for mixed mode fracture separation in viscoelastic media was used. Crack driving forces are then estimated. Due to large differences in the conditions under which temperature and moisture levels can be compared and the variety of wood species, relaxation effects can be observed through the energy release rate evolution versus crack growth process. Then, it was possible to show a general trend, but not a universally valid description to what extent the wood behavior will change at a certain temperature level and what the impact is on the occurrence and development of cracks in wood subjected to thermal and hydrics loadings. In general, it could be concluded that thermo-hydro variation has a greater effect on the reduction of the mechanical proprieties of wood-based materials. However, a critical energy release rate that causes structurer failure and enhance crack growth dramatically, could not be stated, due to the time dependence behavior during wood heating and cooling, but also, no doubt, due to wood species behavior. An experimental procedure is planned in order to validate the numerical analysis. This procedure allows to measure the impact of Thermo-Hydro-Mecanical lodings on the structural collapse. In the end, all data obtained will be compared with results of the numerical model. Comparison of the results will help to refine the analytical and numerical models, and thus to extend their application to other types of wood species such as tropical wood.

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THE EFFECT OF HEAT TREATMENT ON THE WITHDRAWAL CAPACITY OF SCREWS IN FIR WOOD (*Abies borisii-regis*)

Andromachi MITANI
Aristotle University of Thessaloniki, Faculty of Forestry and Environment
Laboratory of Forest Products Technology
54124 Thessaloniki, Greece
E-mail: amitani@hotmail.com

Vasileios FILIPPOU
Aristotle University of Thessaloniki, Faculty of Forestry and Environment
Laboratory of Forest Products Technology
54124 Thessaloniki, Greece
E-mail: vafilpo@gmail.com

Abstract

The present study evaluated the effects of heat treatment at 180^{0}C and 200^{0}C for 3, 5 and 7 in the presence of air in an oven, on withdrawal resistance of three different types of screws for Greek fir wood (Abies Borissi – regis Mattf.). According to the results of the thermally modified samples in comparison with the untreated wood it seems that the screw with the diameter differs statistically at an important level at all treatments except the samples modified at 180^{0}C for 5 and 7 hours. The other two types of screw with the biggest diameter differ statistically significant at all treatments except the samples which are modified at 180^{0}C for 3 hours. Additionaly, the correlation between screw withdrawal capacity and mass loss of heat treated wood is estimated. The dependency between the screw withdrawal capacity and the mass loss was found to be best described by a regression equation with R^2 of 0.97 for the first type of screw, and R^2 of 0.93 for the second and third type of screw.

Key words: *heat treatment; fir wood; withdrawal capacity; screw.*

INTRODUCTION

Wood as biological burning material is influenced and disintegrated under conditions of high temperature. Thermal elaboration of temperature levels of up to 200^{0}C is performed with the main aim of achieving new properties of the material such as changes in color, improved and reinforced biological endurance and increased dimensional stability. The thermal modification of wood could be described as: wood is undergone a process under controlled conditions of temperature, time and in some cases with controlled presence of moisture or oxygen.

This process could have as a result moisture decline, chemical modifications of the polymerical components of wood, alterations in the natural endurance of wood, decline in its hugroscopicity, changes in color and finally in its mechanical strength after a particular combination level of temperature and time (Militz 2002).

Screws and nails are widely used in joining wooden constructions and due to the fact that every kind of wood has different properties, their withdrawal resistance from wood is various. This resistance depends on the density and the specific gravity of wood, the depth of the penetration, the screw or the nail's diameter, the wood endurance, the specie of wood, the moisture content and the temperature exposure of the material and finally the kind of the screw or nail (Ayketin 2008).

Generally the specie of wood with low density restrains nails better than other specie with higher density conception (Soltis 1999). As it has already been mentioned above, the thermal treatment influences the mechanical properties of wood. Therefore, there is great possibility of reducing the withdrawal resistance of the joints. In temperatures above 160^{o}C and specifically above 210^{0}C the withdrawal resistance is seriously decreased due to the mechanical degradation of the elaborated wood, while the treatment duration does not seem to play such an important role in the endurance of the joint. Moreover, the reduced resistance of the joint can be due to the reduction of the shear strength after the thermal treatment. The lower shear strength is combined with the moisture content and density of wood, therefore, it is also combined with the strength of the withdrawal resistance of the joint (Kariz et al. 2013).

Kariz et al. studied the withdrawal capacity of the screw in thermally modified fir sawn wood (Picea abies Karst.) in temperatures 150, 170, 190, 210 and 230^{0}C. According to the results, the withdrawal capacity of screw was reduced with the increase of the temperature and the duration of the

treatment, regardless of the radial and the tangential direction. The statistical analysis showed that the values of the withdrawal capacity for the modified wood differed significantly according to the untreated specimens. The mass and density loss is the main cause of the reduction of the strength. The researchers mention that the distortion of the restraint area of the screw was getting bigger while the temperature and the duration of the treatment increased.

OBJECTIVE

This study was conducted to assess the effect of heat treatment withdrawal resistance of three types of screws of Abies Borissi-regis Mattf. fir wood which is one of the most important commercial wood that is produced in Greece. The correlation between screw withdrawal capacity and mass loss was also investigated.

MATERIAL, METHOD, EQUIPMENT

The investigated material, fir sawn wood (Abies Borissi-regis Mattf.), of Greek origin, was selected from the Greek university forest in Pertouli. These logs were skidded to the sawmill where they were cut into 3.5cm thickness flitches with 15cm width and 50cm length in order to be treated. These flitches were conditioned in laboratory space for about 1 year under 20±2^{0}C and 60±5% relative humidity. The average moisture estimated according to ISO 3130:1975 was 12.27% (SD 0.18) while the average density (oven dry weight/volume at 12.27% (SD: 0.18) moisture) of timber used were 0.40g/cm^3 (SD 0.040).

Subsequently, heat treatment process was conducted in a controlled temperature, small, laboratory heating unit (80x50x60cm) where three different durations (3h, 5h and 7h,) were applied at 180^{0}C and 200^{0}C in the presence of air. The specimens were placed in the unit after reaching the desired temperature. Once heat treating was completed, the specimens were conveyed for air conditioning at 20±2^{0}C and 60±5% relative humidity for 15 days to attain EMC.

The wood samples preparation for the insertion of the screws, the withdrawal resistance procedure and the maximum strength calculation was made based on the standard EN 13446: 2002 (Fig. 1). Three different types of screws were used for the experimentation whose features are: Screw 1: diameter: 2.96mm and length: 60.15mm, Screw 2: diameter: 4.97mm and length: 60.15mm, Screw 3: diameter: 5.80mm and length: 60.15mm.

Fig. 1.
Samples used in tests to determine withdrawal capacity of screws in radial direction.

The dimensions of the wood samples according to the standard are 50mmx50mm. By determining the surface centre of the sample with the use of drilling equipment and creating a hole, the penetration of the screws into the mass of the wood was conducted until the bottom of the screws to exceed at least 1cm from the sample.

The checking procedure of the withdrawal resistance strength was conducted in the strength machine SHIMADZU UH-300kNA where the rate of cross-head movement was adjusted to 5.5mm/min. The moisture content of the samples during the experiment was for the untreated sample: 10.86%, 180^{0}C-3 hours: 9.77%, 180^{0}C-5 hours: 8.02%, 180^{0}C-7 hours: 7.46%, 200^{0}C-3 hours: 6.71%, 200^{0}C-5 hours: 6.30%, 200^{0}C-7 hours: 5.89%.

The results expressed as the maximum load and according to the standard from the equation (1):

$$f = Fmax / d * 1 \quad (1)$$

where:
Fmax: Maximum withdrawal load

d : nominal diameter of fastener
lp : depth of penetration of fastener

One way analysis of variance (ANOVA) comparing the differences of values at 0.05 level was examined in order to determine the significant differences among miscellaneous heat treatment combinations on screws withdrawal capacity.

RESULTS AND DISCUSSION

At Table 1, the mean values of the withdrawal resistance of the different types of screws are presented in Newton as well as the withdrawal capacity per surface unit. As we realize by the results, screw No 1 seems to have increase of its strength regarding the first two treatments at 180^0C for 3 to 5 hours. This fact means that the withdrawal capacity of the particular screw improved after the thermal operation. Accordind to the statistical analysis of the results, the increase that was noticed during the first treatment for 3 hours is statistically significant while the increase in the 5 hours treatment is not considered statistically signifacant. Regarding the other two types of screws the same phenomenon wan not noticed – they showed a slight reduction. Taking the results into account, we can conclude that the withdrawal capacity is reduced in proportion to the temperature increase and to the duration, however, without noticing intense reduction with the change of temperature to 200^0C but only a relatively serious reduction during the last treatment at 200^0C for 7 hours.

In addition, as we can notice at Table 1, the type of screw No 3 shows greater stregth Fmax than the other two types of screws due to bigger diameter of the spiral, except some cases in which type No2 seems to exceed, however, the difference is slght and is certainly unimportant. It is worth mentioning that at the 200^0C treatments the second type of screw shows higher values of withdrawal capacity compared with the third type of screw while regarding the last treatment even the first type of screw shows greater values in comparison with the third type of screw. This phenomenon can be explained because of the friability of the thermally modified woodand the reduction of its density mainly at the intense treatment in which the biggest spiral seems to be operate negatively regarding the withdrawal resistnance of the screw.

The same is not noticed in the untreted wood and is neither noticed at the milder treatment at temperature 180^0C.

Table 1

Strenght and average withdrawal capacity of the screws for wood that was heat treated at different temperatures

Heat treatment conditions	**Units**	**Fir wood- Screw withdrawal capacity of treated and untreated samples**					
		Screw 1(2.96 mm)		**Screw 2(4.97 mm)**		**Screw 3(5.80 mm)**	
		Fmax (N)	**Withdrawal capacity (N/mm²)**	**Fmax (N)**	**Withdrawal capacity (N/mm²)**	**Fmax (N)**	**Withdrawal capacity (N/mm²)**
Untreated	**X**	**1861**	**31,3**	**2349**	**23,54**	**2565**	**21,97**
	±s	76,79	1,31	92,16	0,93	186,33	1,56
	V	0,04	0,04	0,03	0,03	0,07	0,07
	s²	5897	1,73	8494	0,88	3472	2,46
180º C 3 hours	**X**	**2037**	**34**	**2351**	**23,34**	**2339**	**19,96**
	±s	127	2,12	160,23	1,59	189,51	1,61
	V	0,06	0,06	0,06	0,06	0,08	0,08
	s²	1614	4,51	2567	2,55	3591	2,59
180º C 5 hours	**X**	**1942**	**32,59**	**2152**	**21,54**	**2316**	**19,86**
	±s	95,79	1,59	104,15	1,05	131,36	1,1
	V	0,04	0,04	0,04	0,04	0,05	0,05
	s²	9176	2,54	1084	1,11	1725	1,21
180º C 7 hours	**X**	**1821**	**30,54**	**2135**	**21,28**	**2237**	**17,43**
	±s	120,7	1,99	161,64	1,63	97,5	0,85
	V	0,06	0,06	0,07	0,07	0,04	0,04
	s²	1457	3,97	2612	2,68	9507	0,73

200º C 3 hours	**X**	**1765**	**29,52**	**2021**	**20,12**	**2012**	**17,15**
	±s	150,1	2,5	131,4	1,29	95,16	0,78
	V	0,08	0,08	0,06	0,06	0,04	0,04
	s^2	2255	6,29	1729	1,68	9056	0,61
200º C 5 hours	**X**	**1593**	**26,75**	**1927**	**19,22**	**1878**	**16,08**
	±s	118,0	1,93	108,44	1,13	167,73	1,41
	V	0,07	0,07	0,05	0,05	0,08	0,08
	s^2	1393	3,75	1175	1,28	2813	2,01
200º C 7 hours	**X**	**1432**	**23,85**	**1687**	**16,75**	**1415**	**11,99**
	±s	107,1	1,76	149,95	1,41	102,31	0,86
	V	0,07	0,07	0,08	0,08	0,07	0,07
	s^2	1149	3,1	2248	1,98	1046	0,75

* X = average; ±s= standard deviation; s^2=Variance; cv= coefficient of variation

Regarding the modulus of rupture of the three different types of screw, the exactly opposite result is noticed. The modulus of rupture is the stregth of withdrawal per surface unit. Thus, the bigger spiral diameter we have, the smaller the modulus of rupture is. According to the statical analysis of the results, regarding the first type of the screw statistically significant differences are noticed at the 180^0C. Treatments for 3 hours at 200^0C for 7 hours compared with the non-modified wood. In accordance with the results of the thermally modified samples compared with the untreated wood it seems that the first type of screw differs significantly as concerns all treatments ecxept the modified samples at 180^0C for 5 and 7 hours. However, the second and the third type differ significantly at all treatments except the samples modified at 180^0C for 3 hours.

In Figure 2 the mass loss of the samples after heat treatment is depicted and as we can notice, mass loss gradually increased after heat treatment while the duration and time of it were increasing.

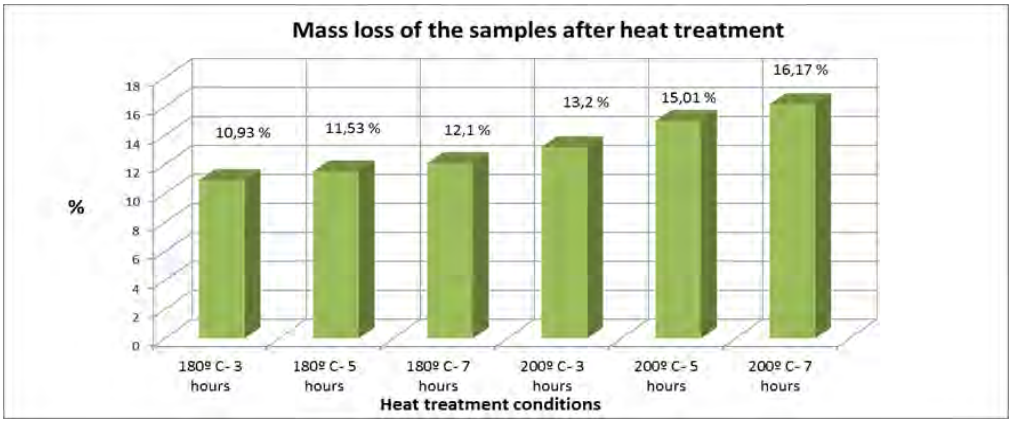

Fig. 2.
Mass loss of the samples after heat treatment.

At figure 3,4 and 5 the correlations between the reduction of the withdrawal resistance of the three different types of screws and the loss of the wood mass after the thermal treatment are shown. According to the correlation level R^2 all three types of screw show a very strong relation with the mass loss. Particularly, the first type of screw showed a relation of 97% (R^2= 0.97) while the second and the third type 93% (R^2= 0.93).

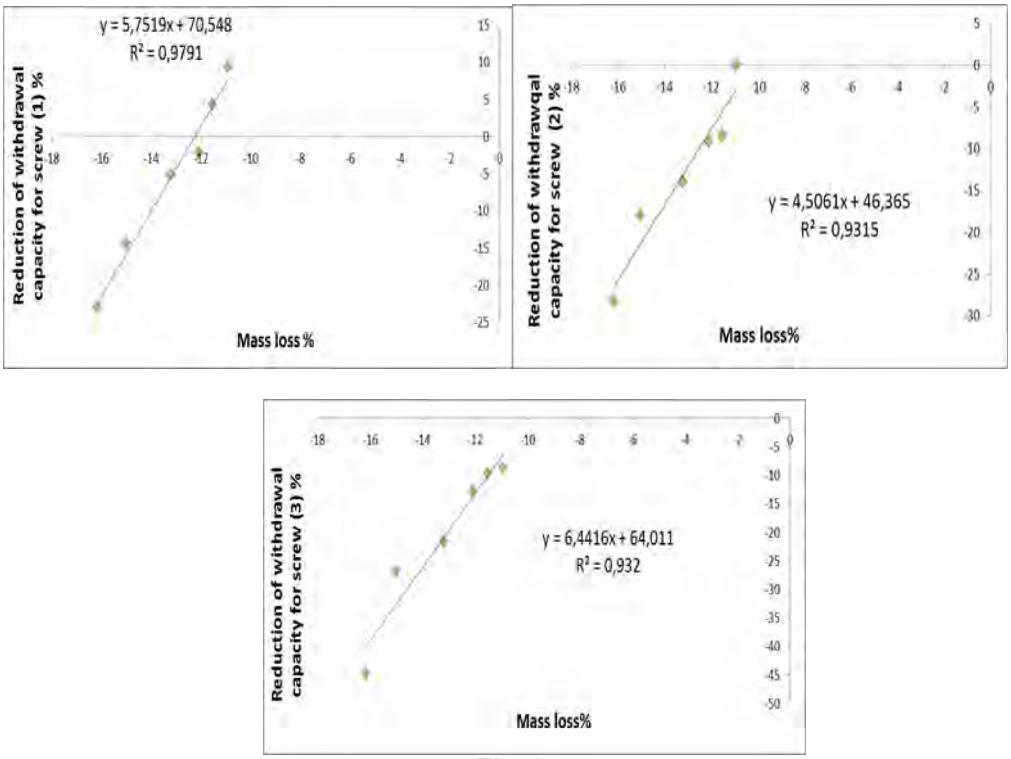

Fig. 3,4,5.
Correlation between screw withdrawal capacity and mass loss.

CONCLUSIONS

Taking into account the results of this research, we reach the following conclusions: Generally, the temperature of the thermal treatment was a more important factor compared with the duration of the treatment regarding the results of the research. The thermallymodified fir sawn wood showed mass loss and the withdrawal capacity of three different types of screws after thermal operation, showed reduction with the temperature increase and the treatment duration except screw No 1 which showed an increase during the first two treatments at 180^0 C for 3 and 5 hours which was 8.62% and 4,1% in correspondence. The withdrawal capacity was more intense regarding the screw with the largest total diameter (thickness) while the screw with the smallest diameter showed weaker strength.The percentance of the reduction of the withdrawal resistance were fluctuated between 20.2% and 23.8% for screw No 1, between 0.87% and up to 28.83% for screw No 2 and between 9.15% and 44.4% for screw No 3.

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ABRASION RESISTANCE OF PINUS WOOD SUBJECTED TO THERMOMECHANICAL TREATMENS

Marina Ulian COELHO
University of São Paulo, "Luiz de Queiroz" School of Agriculture
Avenida Pádua Dias, 11, 13418-900 Piraciba, Brazil
E-mail: marina.ulian@usp.br

Cláudio DEL MENEZZI
University of Brasília, Faculty of Technology, Dept. Forest Engineering
Campus Darcy Ribeiro, 70910-900 Brasília, Brazil
E-mail: cmenezzi@unb.br

Mário Rabelo de SOUZA
Brazilian Forest Service, Forest Products Laboratory
SCEN, Trecho 2, Bl. H, 70818-900 Brasília, Brazil
E-mail: mario.rabelo@gmail.com

Abstract

The study aimed at analising the effect of the thermomechanical treatment on the abrasion resistance of pine wood. Samples measuring 100mm (width) by 100mm (length) by 10mm (thickness) were used for the tests. Six thermomechanical treatments were studied and were compared with wood kept untreated. Two distinct temperatures were chosen for the thermomechanical treatment (160ºC and 190ºC), applying different times, one with pressure (20 minutes) and other without pressure (10 minutes). The densification rates were calculated considering the parameters of thickness (CR) and density (DR). The mass loss was also acessed. The abrasion test was performed in accordance to the ASTM D4060-95 standard, the amount of material lost during the abrasion test was determined by the abrasion rate (AR). It was observed that the higher the temperature the higher de densification rate and the mass loss. The mass loss varied between 11% and 13%, while DRt from 47 to 71%. The highest AR value was observed on the untreated material (0.035%), whereas the lowest on the thermomechanical treatment applied at 190ºC followed by the post-treatment (0.021%). In conclusion, the thermomechanical treatment improved the abrasion resistance of wood from Pinus sp.

Key words: *densification rate; abrasion test; pinus wood.*

INTRODUCTION

The wood from pine is known for presenting low biological resistance, and low resistance to the weathering, therefore, this species usually needs treatments to improve its resistance (Santos et al. 2012). The growing concern with the production quality is justified by the market demands, with customers who are attentive to the products they consume. This forces the industries, and consequently, the suppliers of raw material to improve the quality of the products, in order to attend needs and demands (Vasconcelos and Del Menezzi 2013).

Therefore, the wood densification, associated to elevated temperatures (thermomechanical treatment) is a good alternative, because it improves the physical and mechanical proprieties and also improves the biological resistance and reduces dimensional instability. This kind of treatment improves density and degrades the hemicellulose. During the treatment, the wood losses mass, which might be attributed to the drying and to the partial degradation of their polymers (Santos et al. 2012).

The thermomechanical treatment is an opportunity to make the soft and porous wood denser and turn them useful in situations where greater resistance is needed (Arruda and Del Menezzi 2013). Besides the physical and mechanical proprieties, it is possible to evaluate the resistance of the woods treated thermomechanically regarding the abrasion. The abrasion test consists on simulating real conditions in which the wood will be used. In this case, it consists on trampling of high heels, with small areas of pressure, on dragging and dropping objects, in other words, resisting to the abrasion consists on the friction caused by the displacement of people over the wood (Martins 2008). The resistance to the abrasion is evaluated by measuring the mass variation of a material or the extension of the damage, after submitting it to the abrasive load, during continuous cycles. Due its simplicity, reproducibility and versatility, the Taber equipment is the most used in the world to measure the

resistance to abrasion for several materials (Lopes 2012). Recently, Aytin et al. (2015) used this method to evaluate the abrasion resistance of thermally modified wood.

OBJECTIVE

This research aimed to analyze the effect of the thermomechanical treatments on the abrasion behavior of wood from Pinus sp.

MATERIAL, METHOD, EQUIPMENT

Wood Material

This research was performed at the Sector of Wood Engineering and Physics, at Forest Products Laboratory which belongs to the Brazilian Forest Service (LPF/SFB) and at Laboratory of Engineering and Technology of Forest Products, University of Brasília. Pine wood boards from plantation trees were obtained from the local market and the material was cut into smaller pieces measuring 140mm (width) by 22mm (thickness) by 320mm (length). From this material, samples measuring 100mm (width) by 100mm (length) by 100mm (thickness) were made.

After the visual analysis, the samples that did not present knots, fungus stains or pith were selected, summing 89 samples. Then, the samples were put air-conditining room (20±3ºC; 65±1% RH) up to reach 12% moisture content approximately. Besides, the width, length and thickness of the samples were measured using the digital caliper Mitutoyo Digimatic. Samples were graded according to the density values and divided into seven groups (treatment) with similar initial density: 469-474kg/m^3.

Thermomechanical Treatments

After conditioning, the boards were thermomechanically treated using an automatically controlled single-opening hot press at two temperatures levels: 160ºC and 190ºC. The pressure (25MPa) was applied for 20 minutes (TUP) and was the value corresponding to 50% of perpendicular compression strength. In this step the densification of the sample took place, imparting mass loss and thickiness reduction. After this time, the sample was immediately removed from the hot-press (T1, T3, T4 and T6) while for T2 and T5 the pressure was fully realesed but the sample was kept into the hot-press for further 10 minutes (TWP). However, samples treated under T3 and T6 were treated again one week later again as a post-treatement (PT), as done by Del Menezzi et al. (2006) and Del Menezzi et al. (2009). TWP and PT were considered strategies to reduce springback of the treated wood. The post-treatment consisted on heating the treated samples, at 160ºC/190ºC, for 20 minutes. The same effect was tested for T2 and T5. This way, six different hot-pressing schedules based on the temperature (160ºC/190º), time without pressure (TWP) during compression stage (0´/10´) and time of thermal post-treatment (PT) were evaluated (0´/10´), as can be seen in Table 1. Further detalis about the treatments can be found in our previous paper (Del Menezzi et al. 2015). Table 1 shows the schedules tested.

Table 1

Description of the treatments tested.

Treatments	Temperature [°C]	TUP [min]	TWP [min]	PT [min]
T1	160	20	-	-
T2	160	20	10	-
T3	160	20	-	10
T4	190	20	-	-
T5	190	20	10	
T6	190	20	-	10
Control	-	-	-	-

After performing the treatment and post-treatment, the the compaction rate (CR) was calculated considering the thickness of the samples (Equation 1), while the densification rate according to (Equation 2). Mass loss was calculated according to Equation 3.

$$CR(\%) = (1 - \frac{t_A}{t_B}) \times 100 \quad (1)$$

$$DR(\%) = (\frac{\rho_A}{\rho_B} - 1) \times 100 \quad (2)$$

$$ML(\%) = (\frac{m_B - m_A}{m_B} - 1) \times 100 \quad (3)$$

where:
t_A, t_B: thickness after and before treatment (mm);
ρ_A, ρ_B: density after and before treatment (g/cm³);
m_A, m_B: massa after and before treatment (g);

Abrasion Test

The abrasion test was performed at Sector of Wood Engineering and Physics, at Forest Products Laboratory (LPF/SFB). For the abrasion test, the ASTM D4060-95 standard was followed, which is similar to the NBR 14535:2000. The samples abrasiveness was tested using the Taber (Fig. 1) equipment, with two H18 grinding wheels, at the controlled speed of 60 RPM, a load of 1000 grams was applied in each grinding wheel, with 600 cycles. Aiming to avoid the excessive accumulation of abrasion waste, the grinding wheels were cleaned every 300 cycles. The amount of material lost due the abrasion was determined by the wear rate (WR) as seen on Equation 4.

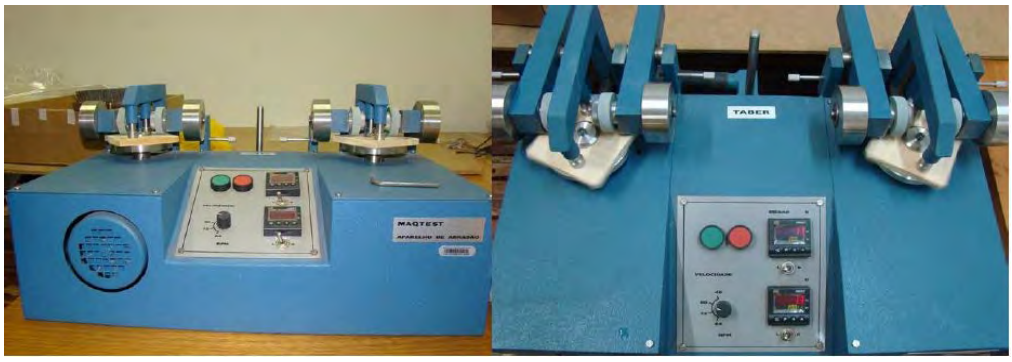

Fig. 1.
Taber equipment used for the abrasion test.

$$WR(\%) = (1 - \frac{t_A}{t_B}) \times 100 \quad (4)$$

where:
WD: wear rate (%);
A: sample's initial mass (g);
B= sample's final mass (g);
C= number of cycles used on the test.

Firstly, the abrasion test was performed on the samples that did not suffer thermomechanical treatment nor post-treatment, it was denominated test-treatment. The control treatment was a parameter to compare the abrasion tests applied on the densified samples. In this step, five samples from each one of the six treatments were chosen. The ones that presented the most regular surface were selected for the abrasion test. Then, eight of the eleven samples from treatment 7 that passed by the abrasion test were submitted to the thermomechanical treatment, with the temperature at 160ºC and pressing time of 20 minutes. Aiming to observe the densification effect on these samples, the abrasion test was repeated.

Experiment Analysis

Initially an analysis of variance (ANOVA) was run followed by the Dunnett mean test, to compare WR values between control and treated material pair to pair. The isolated effect of temperature (160ºC vs. 190ºC) was evaluated by simple F-test. The same procedure was used to evaluate the effect of time without pressure (TWP=T1 x T2, T4 x T5), post-treatment (PT=T1 x T3, T4 x T6) and to compare strategies (TWP/PT= T2 x T3, T5 x T6), as can be seen in Table 2

Table 2

Experiment design to analyse the effect of TWP, PT, strategy and temperature

Effect	Treatment Comparison
TWP	T1 x T2 (160ºC); T4 x T5 (190ºC)
PT	T1 x T3 (160ºC); T4 x T6 (190ºC)
Strategy	T2 x T3 (160ºC); T5 x T6 (190ºC)
Temperature	T1 x T4 (TWP); T2 x T5 (TUP); T3 X T6 (PT)

RESULTS AND DISCUSSION

Variables of the Thermomechanical Treatments

The internal temperature variations for each treatment and for the post-treatment measured by the thermocouple. The curves corresponding to the temperatures 160ºC and 190ºC represent the averages of each treatment. During the initial two minutes of each curve, the temperature gain was quicker. The presence of moisture on the samples enabled the internal heat exchange, providing the accelerated heating on this phase. It is noted in the graphic that the first internal stabilization occurs at 70ºC, highlighting that on the samples submitted to the post-treatment, such stabilization occurred at 130ºC. The total stabilization of the internal temperature occurred on different moments on the three curves observed. The temperature of 160ºC occurred at after 18 minutes from the beginning of the plates heating. As for the time to reach 190ºC was quicker, probably, due the fact that the plates had been heated, previously. During the post-treatment, the 170ºC temperature was reached quickly, at 5 minutes.

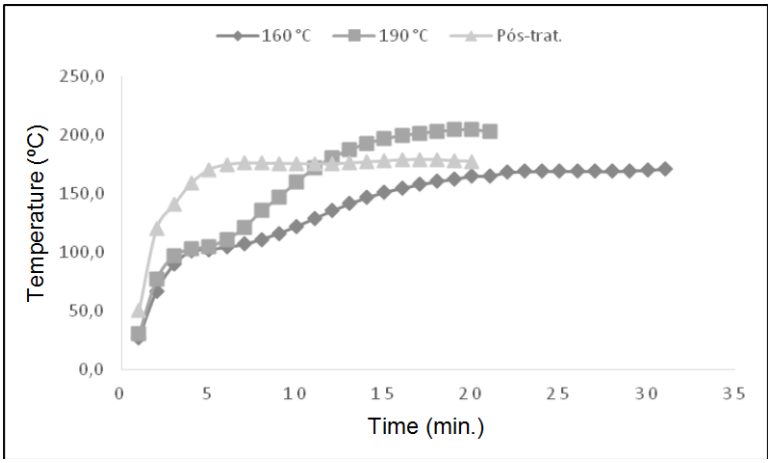

Fig. 2.

Taber equipment used for the abrasion test.

The main effect of the wood compression is the reduction of the spaces between the cells and the cell lumen. Due the heat effect over the wood viscoelastic polymers, the damage on the cell wall occurs without fractures, the vases flatten and the radius get curved (Kutnar et al. 2009). The compaction rates for wood thickness (CR) were higher on T5 (50.6%). The same results could be verified on the wood densification rates regarding density (DR). Again, T5 presented the highest value: 71.2%. Therefore, the use of elevated temperatures reduced the thickness and increased the density. The mass loss varied between 10.9 and 12.8%, being the higher levels verified on treatments with higher temperatures (190ºC). The loss of wood is one of the main characteristics of the

thermomechanical treatments, it is attributed to the drying and partial damage of the wood (Vasconcelos and Del Menezzi 2013).

Table 3

Compaction, densification rate and mass loss of the boards subjected to different thermomechanical treatments

Treatment	CR [%]	DR [%]	ML [%]
T1	45.1	57.3	11.0
T2	45.4	57.3	11.2
T3	41.8	49.6	10.9
T4	49.1	67.7	11.8
T5	50.6	71.2	12.6
T6	49.6	64.5	12.8

According to Del Menezzi et al. (2015) during the heating-compression (TUP) step the hot-press was set to keep the pressure constant. Nevertheless, when the wood is heated the lignin loses stiffness, and passes from a glassy to a rubbery stage. This way, the pressure required to keep the wood deformation goes down (relaxation phenomenon) and thus the hot-press adjusted automatically to the pressure set and further wood densification happened. Fig. 2 shows the appearance of the wood board after the thermomechanical treatments. It is clear the reduction of the thickness which improved the density of the board. Some distortion can be also seen.

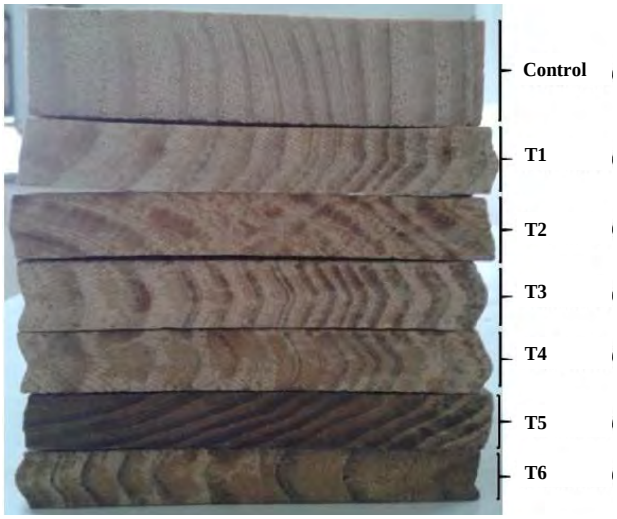

Fig. 2.
View of the thickness and the shape of the board after the thermomechanical treatments.

Abrasion Behavior

The averages wear rates of the abrasion test are represented on Fig. 3. The highest AR value was observed on the control boards (0.036%), while on T6 (190ºC, TWP+PT) the abrasion was significamtly lower (0.021%). However, AR values were not significantly differents when control boards were compared with any other thermomechanical treatment. Aytin et al. (2015) evaluated the abrasion resistance of the wood from wild cherry treated in accordance with ThemoWood process. They assessed the abrasion resistance in terms of weight loss (WL) and thickness reduction (TR) and observed more abrasion in the heated treated boards (tangential) than in the untreated samples.

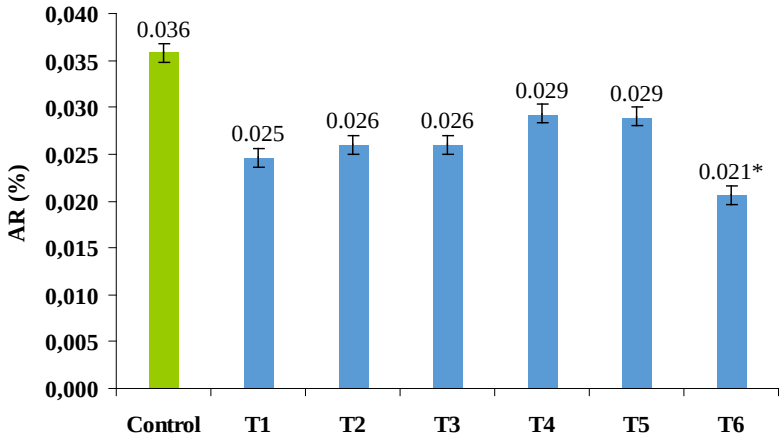

Fig. 3.
Abrasion resistance of pinus wood subjected to different thermomechanical treatments.

Fig. 4.
Apperance of the samples after the abrasion test.

Fig. 4 shows the appearance of the treated boards after the abrasion test. Visually there are not so many differences. According to the ANOVA results (Table 3), for both temperatures keeping the boards into the press or removed imediately led to no differences for AR values (*p*-value 20.6% and 96.8%). When the PT was employed the difference was statistically significantly only for 190ºC (*p*-value 0.008) which means that post treatment one week later was effective to improve the abrasion resistance: 0.021 (T6) x 0.029 (T4). On the other hand, for 160ºC the difference was not significant (*p*-value 0.659). The strategy did not affect the AR values for both temperature tested (*p*-values 0.725 and 0.066). The effect of the temperature was not observed as well (*p*-values 0.133, 0.746 and 0.059). It is unsual results since it is usually known that temperature is the most important factor for any kind of the thermal treatment. In our previous study (Del Menezzi et al. 2015) using the same treatments used here, it was observed that boards treated at 190ºC presented lower equilibrium moisture contente and higher dimensional stability in comparison with those treated at 160ºC.

Table 4

Results of the treatment comparison to evaluate the effect of TWP, TUP, strategy and temperature

Effect	Treatment Comparison	*p*-value
TWP	T1 x T2 (160ºC);	0.206
	T4 x T5 (190ºC)	0.968
PT	T1 x T3 (160ºC);	0.659
	T4 x T6 (190ºC)	0.008
Strategy	T2 x T3 (160ºC);	0.725
	T5 x T6 (190ºC)	0.066
Temperature	T1 x T4 (TWP);	0.133
	T2 x T5 (TUP);	0.746
	T3 X T6 (PT)	0.059

CONCLUSIONS

The thermomechanical treatment improved slighlty the proprieties of the Pinus sp. wood regarding the wear rates caused by the abrasion test. The best treatment observed was at 190ºC and post-treatment. The treatments tested reduced significantly the thickness of the samples, increasing the density.

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INFLUENCE OF FIRE RETARDANT ON SELECTED THERMAL INSULATING MATERIALS ON NATURAL BASE – WOODEN FIBREBOARD.

Linda MAKOVICKÁ OSVALDOVÁ
University of Zilina, Faculty of Security Engineering
Univerzitná 8215/1, 010 26 Žilina, Slovakia
tel.: +421 41 513 6796, E-mail: linda.makovicka@fbi.uniza.sk

Abstract

This paper focuses on flammability of soft wooden fibreboards, which are used as thermal insulation of buildings. The influence of fire retardant and its reaction to fire for selected thermal insulation materials on natural basis – wooden fibreboard treated with fire-retardant – have been tested. Fire retardant has been applied onto the sample by various techniques – spraying, coating and dipping. The paper is divided into five chapters: the first focuses on classification of building materials into fire reaction classes, wooden fibreboards and their fire retardant treatment. The second chapter defines the objective of this paper. The third chapter includes the methodology of the work, description of test samples, testing device and the experiment itself. In the fourth chapter are shown the measured values from two experiments. The fifth chapter is devoted to the evaluation of the values from the experiment and discussion. The result of this paper is comprehensive information about flammability of thermal insulating materials and recommendation of the most suitable way of protecting wooden fibreboard surface using flame retardant.

Key words*: wooden fibreboard; flame retardant; fire reaction classes; insulation material.*

INTRODUCTION

Fiberboard is a large material made from wood fibers of coniferous trees, mostly spruce, or other lignocellulosic materials. This composition, however, has resulted in its inclusion into fire reaction class "E" i.e. to a group of products capable of withstanding a small flame for a short period of time without a significant extension of the flame. The first product resembling hard fibre board was established in England in 1898, but it was hard pressed paper. The first board of wood fibres was produced in 1925. In our region, the production of first fibre board called Hobra was launched after the end of World War II (Östman et al. 2002).

Fibreboard is according to its density divided into:

- Isolated – density 250 - 400 kg.m$^{-3.}$
- Semi-hard - density 480 - 850 kg.m^{-3}.
- Hard - density 850 - 1100 kg.m^{-3}.
- Particularly hard (hardened at a high temperature) - density 1100 - 1300kg.m^{-3}.

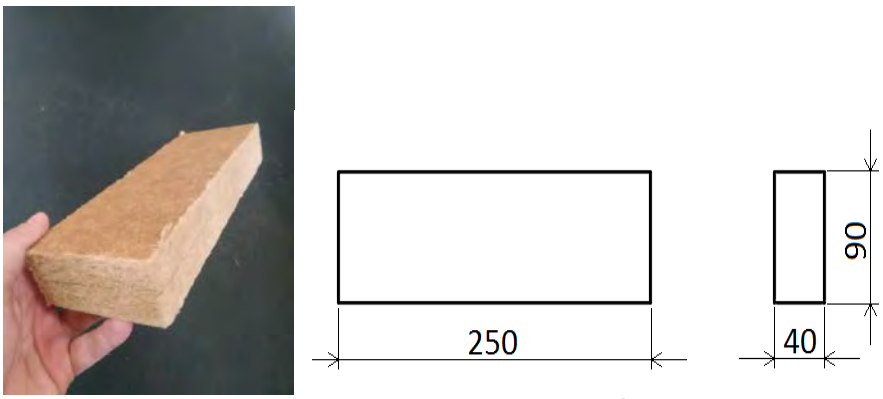
Fibre boards are produced in two different ways - wet and dry. At the beginning of both procedures there are wood chips - free of dirt and cut up. (FEIST 2016) Wet production is energy consuming and more obsolete. Wood fibres are soaked in water, hydrophobic chemical and binders are added into this mixture. The mixture is spread onto a screen where it drains, dries and is pressed. Dry process means that fibre is cut and dried, mixed with adhesive, usually formaldehyde resins and other additives. These fibres are layered and then pressed (Mitterová, Zachar 2013). Soft fibreboard used for thermal and acoustic insulation is usually made using wet process. Adhesives as well as hydrophobic additives, usually paraffin, are added. Pressing takes place under high pressure, resulting in lower strength of hard or semi-hard boards. Therefore, it cannot be used as the sole structural material. On the other hand, it is resistant to anisotropic properties in different ways and has good sound and thermal insulation properties, it is biologically harmless and easy to process. Thermal insulation boards are commonly produced with a thickness of 6-200mm.

FLAME RETARDANTS

Flame retardants are chemical compounds whose chemical, physical, or combined composition protects the material from rapid ignition and subsequent burning. Their main task is to influence those events that result in the cessation of burning. It means a reduction in the rate of heat generation, and increasing the speed of its removal from the reaction zone of combustion. Flame retardants cannot change the intensity of heat flow, but may alter the process and its flammability. This function of flame retardants works in the early stages of a fire, their influence in the later stages is limited since the protected material causes very high heat fluxes. (Gašpercová 2015).

FIBRE BOARD

Soft fibre board (Fig. 1) for the thermal insulation of external walls of buildings, attics, ceilings, roofs and floors was used for the experiment. The material is made of thin and soft wood fibres. Their consistency is ensured by adding glue. Boards were pressed under high pressure. Compared to semi-hard and hard fibre boards, this material has lower strength and cannot be used as the sole structural material. The test samples consisted of two glued layers of wood fibre boards with a thickness of 20mm.



a. b.

Fig. 1.
Fibre board
a - fibre board; b - size of test sample 250x90x40mm.

Test equipment - consisting of a burner, smoke discharger and a sample holder to ensure their stable position - was used for the experiment. The source of ignition is a small propane - butane gas burner. Flame size for all samples was constant - 25mm. The test samples were exposed to the flame of the burner at the angle of 45°. The burner was placed so as to ensure that the flame of 25mm is reaching to the surface 40mm from the bottom edge of the sample. Time was measured using a device with an accuracy of 0, 1s. Weight loss was recorded by a digital instrument with an accuracy of 0.01g. The samples were of the constant size of 250x90x40mm. 5 pieces of the sample were available for each type of fire retardant application.

EXPERIMENT

Fibreboard treated with a flame retardant has been tested. Ohňostop (Firestop) flame retardant (flame retardant based on inorganic salts) was employed. Retarder was applied onto the sample by using various techniques – spraying, coating and dipping (200ml for each sample and each technique). For each application, 5 pieces of samples were available. Marking of the samples was as follows: B1-5 non – treated sample, S1-5 spraying, N1-5 coating, M1-5 dipping (for second test, we tested the samples M 6 – 20 for dipping only). After the application, we reweighed the samples and recorded their weight gain after the treatment. This procedure was repeated with all technological processes (dipping, spraying, and coating).

The samples were drying for seven days and reweighed afterwards. Each sample was then marked by a line 40mm from the lower and upper edge which determined the maximum upper limit of the flame. Before testing, total weight of the dried samples was recorded again. The samples were then mounted to the test device and exposed to the open flame of the burner for 30 seconds. During the measurement, the behaviour of the samples was observed - creation of smoke and the time when the flame exceeded the upper line. Finally, the weight of each sample determining the weight loss - was measured. Weight loss of the test samples - calculated according to following equation - was the evaluation criterion (1):

(1)

$$\Delta m = \frac{m_1 - m_2}{m_1}.100$$

where: Δm is the weight loss (%),
m_1 is the sample's weight before the test (g),
m_2 is the sample´s weight after the test (g).

Since none of the applications did not show significantly better results compared to untreated samples, we decided to carry out an additional measurement. Based on the previous tests, we found out that dipping was the best application process. Therefore we wanted to determine whether soaking time of the specimen in flame retardant solution will also affect its reaction to fire. After dipping, the fiber board was soaked into the fire retardant for 6, 9, and 12 minutes, the amount of retardant the material could absorb was monitored. (Fig.2) (Marienka 2016)

Fig. 2.
Fiber board after dipping
a – pre-test, b- dipping after 6 min, c – dipping after 9 min, dipping after 12min.

RESULTS AND DISCUSSION

The main evaluation criterion was the weight loss of fire retardant treated test samples and the possible formation of smoke and burning particles. Based on the measured values, we found out that the surface protection of fire retardant treated fibreboard can be moved from the fire reaction class E into fire reaction class D. Another criterion was to determine the most appropriate technological process of fire retardant application (dipping, coating, spraying). Considering the measured results, we can conclude that the best technique for the application of flame retardant onto fibreboard is soaking at a time interval of at least nine minutes. With the given data, we were able to propose a number of recommendations for practical purposes, either further testing with different types of flame retardants such as the latest flame retardants based on nanotechnology, but mainly natural based retardants so as to maintain unity by using natural materials. (Fig.3)

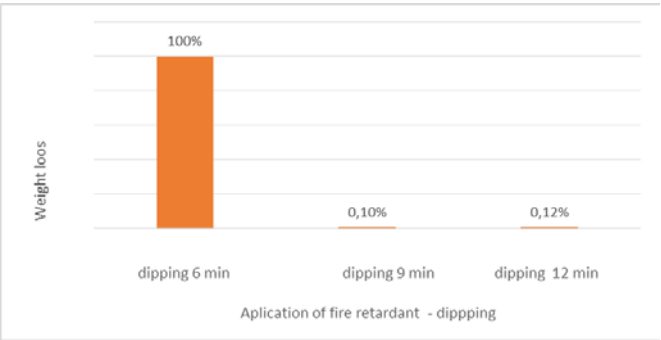


Fig. 3.
Various techniques of fire retardant application (dipping).

Based on the measurements, we came to the following conclusions: Among all flame retardant applications (coating, spraying and dipping), only dipping seemed to withstand fire exposure. Spraying and coating failed the test, as the flame disrupted the protective layer formed by flame retardants and the degradation of the inner structure of the fiber board led to complete decay of the material. These results were observed with all the tested samples and flame retardant applications.

Smoldering of the samples was quite difficult to extinguish since smoldering constantly renew until the boards were not completely soaked in water. The samples dipped in flame retardants for three minutes failed the test (Fig. 4). The only exception was the sample M2, which was the sole one withstanding the flame exposure. (Marienka 2016)

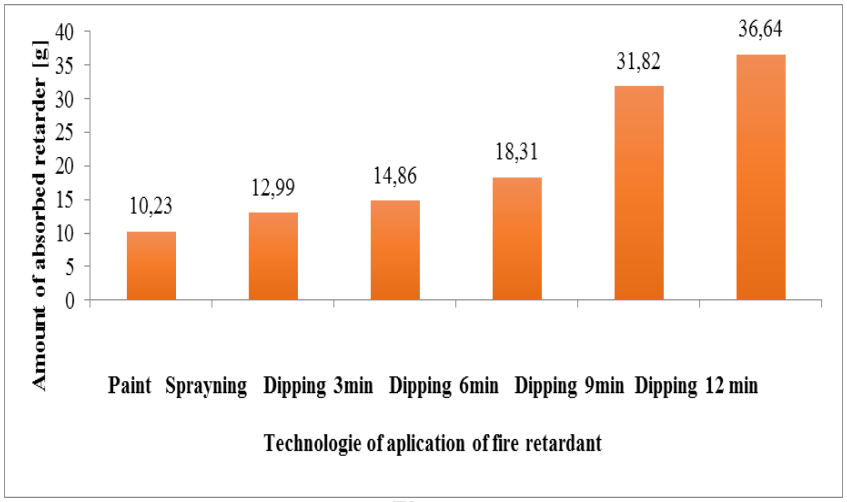


Fig. 4
Various technique of retardant application

Based on the improved results, we made additional measurements in order to determine whether longer soaking time affects fire resistance of the material.

In the second experiment, we tested the samples soaked in flame retardant for 6, 9 and 12 minutes. The length of soaking significantly increased the amount of flame retardant absorbed. During the 6-minute interval, the sample absorbed 18.31g of the flame retardant on average, 31.82g after 9 minutes and 36.63g after 12 minutes. Out of the five specimens, only two (M7 and M8) were measured for the 6 minute dipping test. Their weight loss was minimal - 0.06 and 0.09% of the total weight of the sample. This weight loss was caused by scorched surface of fiber board – the inner structure of the samples was not damaged, not even the point of greatest flame exposure. Other samples did not pass the test.

The desired results were achieved with the samples dipped in Firestop solution for 9 minutes. All of the samples tested met the requirements. This result was achieved thanks to the amount of flame retardant absorbed. From all of these samples, the sample which absorbed the least amount of fire retardant was the one labeled M14 - 27.43g of the solution. This represented 16.02% of the total weight of the sample just after soaking. This amount of flame retardant created a salt layer on the surface of the material, preventing the flame from penetrating the board. Weight loss ranged from 0.03 to 0.15%. The average weight loss was 0.10%.

Longer soaking time caused that the sample absorbed from 34.22 to 42.68g of the solution. The only exception was the sample M17 which - because of its composition - absorbed 26.03g of the flame retardant. This represents 15.47% of the weight of the sample immediately after application. This sample did not withstand the test, its weight loss was 100%. Other samples showed a weight loss of 0.10 to 0.16%. The test samples lost 0.12% of its original mass on average. The relationship between the absorbed amount of the flame retardant and the % of weight loss for the sample is represented by the following graph (Fig. 5).

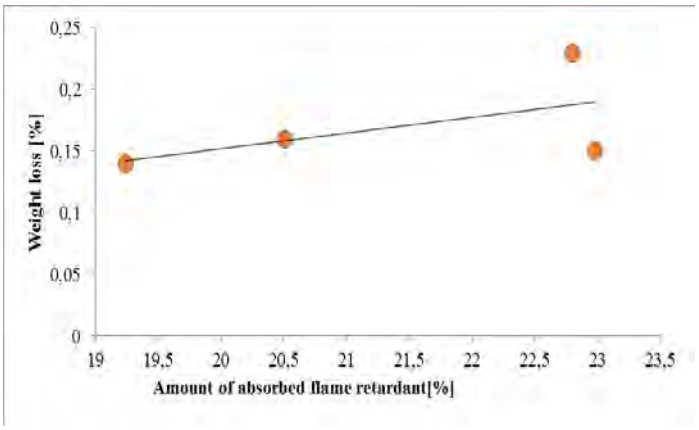


Fig. 5.
Graph of the amount of absorbed retarder in percentage and its weight loss after 12 minutes of soaking.

CONLUSIONS

Based on the results, we can conclude that the most appropriate method of applying the flame retardant is dipping. In our case, it was necessary to soak the samples in Firestop solution for at least 9 minutes to get reliable results. If we wanted the samples to pass the test, each sample had to absorb at least 16, 02% of the sample weight immediately after flame retardant application. Another observation was that the flame does not burn on the surface, but thanks to thermal degradation of the board the burning continues even after turning off the flame source. Attempts to extinguish the test specimens have been unsuccessful several times despite having applied an extinguishing agent (water) firmly, smoldering within the material tend to restore. In one of the tests, the sample smoldered internally and the entire length of the sample was burnt through. We managed to extinguish the samples when immersed into water for a few seconds. As a result, the water permeated the inner layers of the board and interrupted the smoldering.

This poses problems for intervention units. In case of a real fire, there is a risk of fire recurrence or its hidden paths. The problem may also be the amount of water needed to extinguish the fire and the subsequent damage it has caused. Adding flame retardant additives in the production process might solve the problem. That way, the surface as well as individual fibers and thus the inner structure of the board would be protected. The question is – would the change affect the other properties of the material such as mechanical strength, and vapor permeability?

Another problem was the amount of smoke produced during smouldering. This poses additional problems for rescue services having to use protective equipment. In practice, there are flame retardants of different chemical compositions, some of which are toxic to man. In case of a fire, the release of these fumes are a threat to rescue services. Since the presence of such substances is not immediately apparent, adding such data into database might represent the solution. The data would be provided by the operations chief or by physical identification represented by labels used directly on the site.

According to the given data, a number of recommendations for practical purposes were proposed – either it is tests of different types of flame retardants such as the latest flame retardants based on nanotechnology or retardants on natural basis so as to maintain the unity using natural materials.

ACKNOWLEDGMENTS

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EFFECT OF THERMAL MODIFICATION ON MECHANICAL PROPERTIES AND SURFACE ROUGHNESS OF POPLAR WOOD

Vasiliki KAMPERIDOU
Aristotle University of Thessaloniki, Faculty of Forestry and Natural Environment
Laboratory of Wood Products and Furniture Technology
54124 Thessaloniki, Greece, E-mail: vkamperi@for.auth.gr

Ioannis BARBOUTIS
Aristotle University of Thessaloniki, Faculty of Forestry and Natural Environment
Laboratory of Wood Products and Furniture Technology
54124 Thessaloniki, Greece, E-mail: jbarb@for.auth.gr

Abstract

Thermal modification of poplar (Populous sp.) wood was implemented at 180°C and 200°C, for 3, 5 and 7 hours in the presence of air and some of the most crucial mechanical properties were investigated. As the intensity of heat treatment increases, the mass loss of poplar boards is increased, attributed to moisture loss and the degradation and loss of chemical wood components. The bending strength decreased (2.05%-44.65%) compared to control, due to the treatments, while the elasticity decreased in all the treatment categories (5.66%-24.86%). The tangential hardness of poplar modified at 180°C for 3 and 5 hours, decreased by 10.24% and 14.59%, respectively and at 7 hours it grew by 2.35%, while at 200°C a decrease of 17.04 -26.05% was recorded. The radial hardness decreased (2.81%-11.06%) at 180°C (3 and 5 hours) and when the duration reached 7h, it increased (9.28%), while at the treatments of 200°C radial hardness decreased (2.68%-18.86%). All the treatments reduced the impact bending strength of poplar specimens (24.27%-62.02%), even though they raised the resistance levels to compressive forces. Only the milder treatment achieved to decrease the roughness of poplar wood surface, while increasing further the treatment intensity, the degree of roughness showed a strong increase.

Key words: *mechanical properties; poplar; thermal modification; treatment; roughness.*

INTRODUCTION

The history of thermal treatment of wood, aiming at improving its properties, changing the shape or form, but also increasing its life duration, begins prior to several thousands of years. The results of the heat impact on improving dimensional stability and biological resistance of wood were already known several years ago. High temperature causes chemical changes in the wood components of large molecular weight, thus altering the physical and biological properties of it.

As the temperature increases, chemical changes are conducted in the components of the cell walls, resulting in further reduction of weight of the sample, and this mass loss corresponds to change of several properties, which is the result of the first stage of wood kinetic processes. Each type of wood reacts in a different manner to heat treatment, while large differences appear between softwoods and hardwoods. Researches have shown that the larger mass losses are presented in hardwoods, either referring to heat or in hydrothermal treatment (containing larger proportion of hemicelluloses) (Zaman *et al.* 2000). In each wood species the same moisture-heat interaction mechanisms take place, but the moisture transfer rate is different, because of the structure and porosity (Younsi *et al*. 2010). Additionally, the dimensional stability of wood is reduced, as well as the hygroscopic nature of wood (shrinkage-swelling), including the decrease in equilibrium moisture content (EMC) and wettability (Gonzalez-Pena *et al.* 2009, Ates *et al.* 2010).

Heating of wood at a temperature till 100°C, its mechanical properties decrease, but the effect is not permanent, while it begins to become permanent when the heating is at temperatures higher than 100°C, depending on the duration and moisture content of wood. Thermal modification at elevated temperatures has been proved to cause a reduction in mechanical strength, especially impact bending, tensile and flexural forces, but also reduces hardness, abrasion resistance and other properties (Hill 2006, Diouf *et al*. 2011). Acetic acid and other by-products of thermal degradation seem to help reduce the mechanical strength of the modified wood and as the temperature and the treatment duration increase, the thermal degradation is intensified. This has led to the development of thermal modification methods using temperatures till 200°C.

In Greece, the species of *Populus alba, P. tremula, P. nigra* and *P. canescens* grow, and mainly hybrid plantations appear in the region of Macedonia, Thrace and Thessaly. Indicatively, in 2011 the production of public and private forests in Greece in Populus was 13003, 10829 and 3783 m3 (total: 27616m^3) for technical, industrial and fuel wood, respectively (Ministry of Environment 2014). The wood of this species is of low quality, mechanical resistance and is susceptible to microorganisms attacks, therefore, the application of thermal treatment seem to be necessary, in order to improve some of its properties and increase its duration of use.

OBJECTIVE

Objective of this specific research work is to investigate the influence of thermal modification at the temperature of 180^{o}C and 200^{o}C, for the duration of 3, 5 and 7 hours on the mechanical properties and surface roughness of a fast growing wood species, such as poplar (Populous sp.) wood, in order to look into the possibility of improving some of the properties of this species, widening in this way the future range of its applications and uses.

MATERIAL, METHOD, EQUIPMENT

Boards of poplar wood (*Populus* sp.) obtained from plantations of North Greece (Drama) were transferred to the laboratory of wood science in the Faculty of Forestry and Natural Environment (AUTH), cut parallel to grain and placed for approximately 8 months in a climate chamber (20±2°C and 60±5% RH), until a constant weight, where an equilibrium moisture content (EMC) of 10.50% (0.521 standard deviation) was acquired. This is a relatively low level of moisture content, in order to avoid the creation of internal stresses and thus distortions of wood during the treatment process. The mean density of poplar before thermal treatment (oven dry weigh/volume in moisture content level mentioned) was 0.385g/cm^3 (0.02 St. dev.). The dimensions of the plates intended to participate in heat treatment was 35mm thickness x 70mm width x 400mm length.

The thermal treatment of wood took place in a laboratory drying chamber (80cm x 50cm x 60cm), where the treatment was conducted at temperature of 180°C and 200°C, under atmospheric pressure conditions in the presence of air, while the chamber was pre-heated at the final temperature. The treatment lasted 3, 5 and 7 hours (counting 15 additional minutes each time for the recovery of temperature inside the chamber) and 10 plates were used in each treatment. According to the literature, the specific temperature levels and durations of treatments have not been tested so far. At the end of treatment duration, the plates were placed in glass desiccators to return gradually to ambient conditions and subsequently stacked in a conditioning chamber of stable conditions (humidity 60±5%, 20±2°C).

Before heat treatments, measurement of weight and exact dimensions of each of the plates was performed, just as it was measured directly after the treatments, in order to record the mass loss of wood due to the process. Weight and dimensions of plates were also measured 1, 2, 3 and 4 weeks after treatment, in order to detect the recovery rate of moisture of the plates during their conditioning.

The plates of treated and untreated wood were cut to final dimensions for the properties testing, according to relevant standards and the final samples were conditioned for 1 month more till their constant weight was achieved. For each variable, 15 specimens were prepared.

After the conditioning of the samples, EMC and the density of wood was determined again using the standards ISO 3130:1975 and ISO 3131:1975.

The resistance tests to static bending forces, as well as the elasticity tests, were conducted in accordance to standard ISO 3133:1975 in Universal Testing Machine, with a loading piston speed of 5mm/min. The loading was applied tangentially to specimens in the middle of their length.

The standard on which the test of the tangential and radial «Janka» hardness of the specimens was based, was ISO 3350:1975, using an Amsler Universal Testing machine and applying a piston speed of 6mm/min. The resistance test of the specimens to impact bending forces was conducted according to ISO 3348:1975 standard, in Amsler Machine, by performing loading at the center of each specimen, perpendicularly to the tangential surface of the samples. The compression test of the specimens was conducted according to DIN 52185:1976, in the same machine, with a piston speed of 6mm/min.

The surface roughness of the samples was determined using a profilometer apparatus (Mitutoyo Surftest SJ-301) and a diamond stylus device. The measuring speed, the pin diameter and the top corner of the pin tool was 10 mm/min, 4μm, and 90^o, respectively. The roughness indexes values were determined with an accuracy of ± 0.01μm. The points of roughness measurement were randomly chosen on the surface of the samples. Measurements were implemented in a direction perpendicular to the fiber direction of the samples. Three roughness parameters were recorded, the mean arithmetic deviation of profile - Ra, the mean peak-to-valley height - Rz, and the root mean

square deviation of the assessed profile - Rq (ISO 4287, DIN 4768). All the plates were subjected to sanding before the measurment and then, cut to smaller dimensions (50mm x 50mm x 50mm) for the roughness test, which was carried out in the tangential surface, because it usually presents lower roughness, compared to radial one (Budakci *et al*. 2013). For the statistical analysis of the results, SPSS Statistics PASW was used in order to analyze the variability of mean resistance values using the method Bonferoni and Tamhane (One way ANOVA), as well as the Least Significant Difference method (LSD - two-way ANOVA).

RESULTS AND DISCUSSION

According to the results (Table 1), as the treatment intensity, referring to the temperature and duration, increases, the rate of mass loss of the specimens is increased due to the processes taking place during wood modification and can be attributed to the moisture loss, the evaporation of volatile extracts, the loss of chemical constituents due to their degradation, evaporation and loss of thermal degradation products (mainly hemicelluloses) from the mass of wood (Kocaefe *et al.* 2008).

Table 1

Average mass loss percentage values of poplar specimens after heat treatment

	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
Mass Loss	**11.237 (2.512)**	**11.601 (0.645)**	**11.935 (0.975)**	**13.461 (2.192)**	**16.409 (1.974)**	**18.884 (2.642)**

standard deviation values within the brackets

Corresponding researches has shown that hardwoods present larger mass losses compared to conifers, probably due to the higher percentage of hemicelluloses (pentoses), which are more susceptible to thermal degradation and already depolymerized at 180-200°C. Additionally, hardwoods have a slightly lower lignin content, which seem to be more resistant to thermal degradation than the other components (Gonzalez-Pena *et al.* 2009).

According to statistical analysis, all the mean loss values of the samples were found to differ statistically significantly from one another, except the cases between control and the two milder treatments (180°C -3 and 5 h) and also the case between samples of treatment of 180°C (5 and 7h).

Significant mass loss, proportional to the treatment temperature, was recorded by corresponding investigations: Esteves *et al.* 2007, Korkut and Budakci 2010, Schneid *et al*. 2014 etc.

There was a decrease in the recovery rate of the initial weight of the specimens, which is more pronounced as the treatment temperature and duration increase (Table 2). This means that the EMC obtained by the samples after their conditioning tends to stabilize at lower levels than those of control, and as is evident, the modified samples of shorter duration treatments exhibit higher weight increase, due to higher absorption of moisture from the surrounding atmosphere, compared to the samples of more intensive treatments.

Table 2

Conditioning progress (moisture recovery rate) of boards indicated by values of weight gain percentage 7, 14, 21 and 28 days after treatment

Treatment	Weight increase % (7 days)	Weight increase % (14 days)	Weight increase % (21 days)	Weight increase % (28 days)
180°C-3h	**2.533 (0.335)**	**3.057 (0.336)**	**3.532 (0.371)**	**3.983 (0.389)**
180°C-5h	**2.427 (0.350)**	**3.066 (0.393)**	**3.523 (0.457)**	**3.932 (0.478)**
180°C-7h	**2.509 (0.405)**	**2.958 (0.419)**	**3.355 (0.395)**	**3.727 (0.397)**
200°C-3h	**2.125 (0.312)**	**2.730 (0.384)**	**3.142 (0.498)**	**3.585 (0.462)**
200°C-5h	**2.073 (0.333)**	**2.586 (0.301)**	**2.865 (0.319)**	**3.349 (0.272)**
200°C-7h	**2.033 (0.196)**	**2.704 (0.253)**	**2.992 (0.249)**	**3.412 (0.227)**

Standard deviation values within brackets

As the treatment intensity increases, both EMC and density of wood are reduced Table 3). EMC of specimens, modified at 180°C for 3, 5 and 7 hours presented a decrease of 18.76%, 24.12% and 28.42%, compared to control, whereas those modified at 200°C for 3, 5 and 7 hours, a decrease of 41.17%, 45.83% and 49.20%. This reduction in EMC clearly indicates that thermal treatment greatly affects the dimensional stability of wood, the absorption of moisture from the atmosphere and is directly related to the reduction of hydroxyls in wood mass due to treatment.

Table 3

Mean values of EMC and density of treated and untreated specimens after 4 weeks of conditioning

Treatment	EMC %	Density (g/cm^3)
Control	**10.506 (0.521)**	**0.385 (0.002)**
180°C - 3h	**8.535 (0.377)**	**0.381 (0.006)**
180°C - 5h	**7.972 (0.500)**	**0.356 (0.007)**
180°C - 7h	**7.520 (0.871)**	**0.346 (0.007)**
200°C - 3h	**6.181 (0.654)**	**0.336 (0.005)**
200°C - 5h	**5.691 (0,246)**	**0.333 (0.002)**
200°C - 7h	**5.337 (0.402)**	**0,291 (0.005)**

Standard deviation values within brackets

The samples modified at 180°C for 3, 5 and 7 hours showed a decrease in density of 1.04%, 7.53% and 10.13%, respectively, while the 3, 5 and 7 hours treatment at 200°C, a decrease of 12.73%, 13.51% and 24.42%, compared to control. Schneid *et al.* (2014), Günduz and Aydemir (2009), Esteves *et al.* (2007), Arnold (2010), Ates *et al*. (2010), Niemz *et al.* (2010) etc. have presented also similar results and behavior of thermally treated wood. This density decrese is also associated to the reduction in moisture content after treatment and the mass loss caused by the thermal decomposition of hemicelluloses and the evaporation of volatile wood extracts that leave vacant places in its mass. Wood density decrease is also associated to a potential of mechanical properties decrease, though the decrease of EMC contributes to the increase of mechanical strength (Hill 2006).

Thermal treatment at 180°C for 3, 5 and 7 hours seem to have decreased also Modulus of Rupture (MOR) by 2.05%, 7.62% and 21.89%, respectively. An even stronger decrease of MOR was marked in specimens that underwent treatments of 3, 5 and 7 hour at 200°C, revealing a decrease of 32.04%, 34.56% and 44.65%, compared to control (Fig. 1). Two Way ANOVA method applied to MOR values of the samples showed that temperature is a very important factor, which has a statistically significant effect on MOR affecting by 77% its variability.

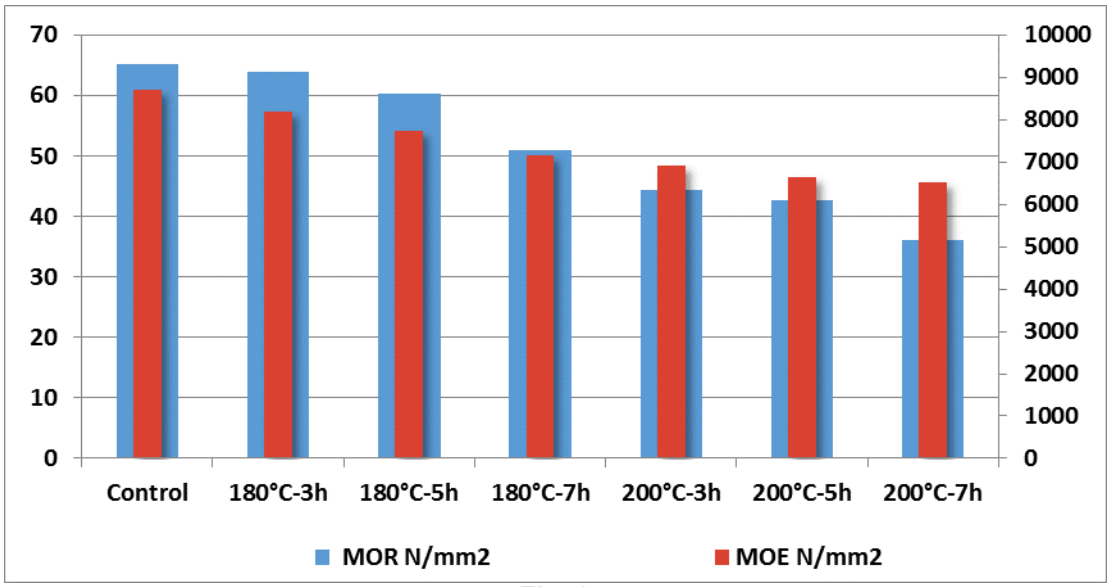

Fig. 1.
Mean Modulus of Rupture and Modulus of Elasticity values (measured in the bending test process) of the control and thermally modified specimens (N/mm^2).

Heat treatments of 180°C for 3, 5 and 7 hours showed a 5,66%, 11,19% and 17,57% decrease in the modulus of elasticity (MOE), while the treatment of 3, 5 and 7 hour at 200°C recorded a 20.53%, 23.67% and 24.86% decrease. The factors of temperature and time have a statistically significant effect on MOE, while the temperature affects its variability by 26% and the time by 10.4%. Kocaefe *et al.* (2008) reported a relatively slight increase in MOR and MOE of Canadian birch and poplar at 160-200°C, probably due to the branching of lignin in wood, while they both decrease to temperatures above 200°C. Most researches, however, report a decrease in MOR and a slight increase in elasticity (Olek and Bonarski 2008, Li Shi *et al.* 2007, Németh and Miklós 2012, Goli *et al.* 2014, Akyildiz *et al.* 2009).

According to hardness tests results, all thermal treatments, except for 180°C-7hours, decreased hardness of wood in both tangential and radial cross-section (Table 4). More specifically, the tangential hardness of specimens, modified at 180°C for 3 and 5 hours, decreased by 10.24 and 14.59%, while by increasing the duration to 7 hours the hardness increased slightly (by 2.35%), compared to control. This increase may possibly be attributed to the formation of some components in woody mass, which probably enhances wood hardness. Samples treated at 200°C for 3, 5 and 7 hours presented reduced tangential hardness by 17.04%, 24.92% and 26.05%, respectively.

Table 4
Mean tangential and radial hardness values of thermally treated and untreated specimens (kN)

Hardness (kN)	Control	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
Tangent.	1.954 (0.221)	1.754 (0.174)	1.669 (0.157)	2.000 (0.285)	1.621 (0.183)	1.467 (0.118)	1.445 (0.129)
Radial	2.025 (0.213)	1.968 (0.189)	1.801 (0.157)	2.213 (0.262)	1.972 (0.161)	1.710 (0.170)	1.643 (0.139)

Standard deviation values within brackets

Regarding the radial hardness of the specimens, it decreased by 2.81% and 11.06% in the mildest treatments (180°C for 3 and 5h), while at 7 hours treatment it increased by 9.28%. Samples treated at 200°C for 3, 5 and 7 hours revealed a reduce of 2.68%, 15.52% and 18.86%, respectively. In a similar research by Li Shi *et al.* (2007), thermally modified poplar showed a decrease in radial, tangential and transverse hardness by 26%, 39% and 15%, respectively, while Goli et al. (2014) recorded similar findings, as well.

The effect of temperature on the tangential hardness of poplar wood was statistically significant and the main factor affecting its variability by 39.6%. A statistically significant effect was

also found in the temperature-duration interaction, in which 19.9% of the hardness variability is attributed, while the duration affects it only by 11.3%. Similar were proved the two-way ANOVA findings for the radial hardness.

Heat treatment at 180°C for 3, 5 and 7 hours reduced the impact bending strength by 24.27%, 37.56% and 47.40%, respectively, while treatments of 200°C resulted in a strength decrease of 45.73%, 49.54% and 62.02%, compared to control (Table 5). Generally, the intensity of the particular thermal treatments applied in the present project was of such intensity, that caused a high decrease in the impact bending strength of poplar.

Table 5

Mean values of impact bending strength (J/cm^2) of heat-treated and untreated specimens

Impact Bending (J/cm^2)	Control	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
	2.620 (0.312)	1.984 (0.395)	1.636 (0.290)	1.378 (0.186)	1.422 (0.177)	1.322 (0.153)	0.995 (0.086)

Standard deviation values within brackets

Temperature and time appeared to affect the variability of impact bending strength (46% and 46.5% respectively), both of which had a statistically significant effect on impact bending resistance. The interaction between temperature-time can be characterized as not statistically significant.

According to compression test results, heat treated samples showed higher levels of resistance than control levels (Fig. 2). Even the milder treatment (180°C-3h) was sufficient to bring about a satisfactory improvement in the compression strength of poplar specimens, and as treatment duration increases (at 180°C), strength increases as well. Specifically, treatments of 180°C (3, 5, 7h) increased the compression strength by 13.70%, 15.84% and 18.14%, respectively, while treatments of 200°C (3, 5, 7h) resulted in an increase of compression strength by 23.26%, 23.18% and 22.13%, compared to control. Németh and Miklós (2012), as well as Mazela *et al.* (2010), also recorded a slight increase in compressive strength of poplar wood after thermal treatment.

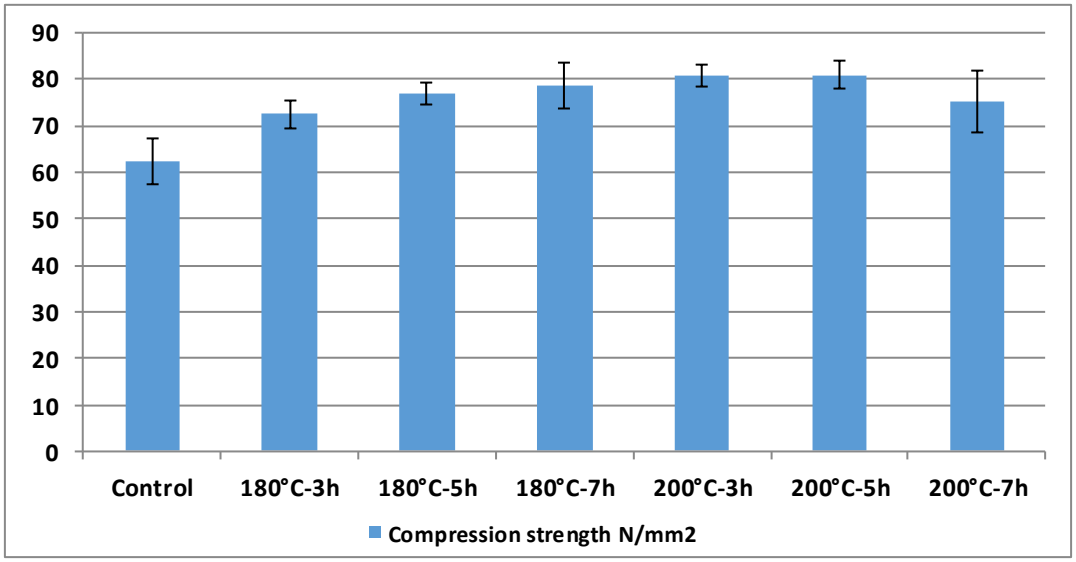

Fig. 2

Mean values of compression strength (N/mm^2) of heat-treated and untreated poplar samples.

According to statistical analysis, only the compression strength of control specimens was found to differ significantly from all the modified categories of specimens. Temperature was found to be the major factor affecting the compression strength variability by 17.1%.

According to the roughness tests on the surface of poplar specimens, it emerged that only the mildest treatment slightly reduced the roughness of wood surface, while increasing the temperature and duration of the treatment, the degree of roughness showed an intense increase compared to control levels. Specifically, the roughness index Ra value (most representative) of the specimens treated at 180°C for 3 hours, presented a slight decrease of 2.80%, while as the temperature and the

duration increased, Ra showed an increase of 26.92% to 70.36%, compared to control level. This behavior may be attributed to the fact that thermal treatment causes a mass loss, making wood more brittle, reducing the number of hydroxyls and its moisture content, causing degradation of the hemicelluloses and all these affect the roughness of wood surface (Budakci *et al.* 2013). In contrast, in several previous researches (Korkut and Guller 2008, Korkut *et al.* 2008, Korkut and Budakci 2010, Schneid *et al.* 2014, Baysal *et al*. 2014) the surface roughness was found to be decreased as the temperature and duration of thermal treatment increases.

Table 6

Mean values of the surface parameters (Ra, Rz, Rq) of thermally modified and control specimens

Parameter	Control	180°C-3h	180°C-5h	180°C-7h	200°C-3h	200°C-5h	200°C-7h
Ra	2.355 (0.347)	2.289 (0.181)	2.989 (0.235)	3.137 (0.314)	3.150 (0.304)	3.128 (0.384)	4.012 (0.302)
Rz	18.106 (1.772)	17.366 (2.455)	22.225 (2.396)	24.528 (1.689)	22.913 (1.852)	23.348 (2.527)	27.541 (1.885)
Rq	3.225 (0.449)	2.965 (0.223)	4.005 (0.339)	4.403 (0.433)	4.205 (0.291)	4.211 (0.500)	5.227 (0.314)

Standard deviation values within brackets

According to statistical analysis of Ra roughness values, the effect of temperature and time factors was found to be both statistically significant, affecting its variability by 55.5% and 61.2% for temperature and duration, respectively. The interaction between temperature-time was also statistically significant, affecting the variance of Ra by 27.4%.

CONCLUSIONS

According to the research, as the treatment intensity increases, higher mass losses, caused by the treatment, are being recorded, which corresponds to moisture and the wood components loss, due to thermal degradation. All treatments resulted in a reduction of the recovery rate of the initial weight, due to the reduced moisture recovery, which is more intense as the temperature and duration of treatment increases. EMC of all the samples was found reduced, compared to control, even in the case of the mildest treatment, while as the treatment intensity increases, a greater decrease is marked (18.76% -49.20%). Additionally, The bending strength decreased (2.05%-44.65%) compared to control, due to the treatments, while the elasticity decreased in all the treatment categories (5.66%-24.86%). Furthermore, the tangential hardness of poplar samples modified at 180°C (3, 5, 7h), declined by 10.24%, 14.59% and an increase of 2.35% in 7 hours, while treatments of 200°C reduced the tangential hardness by 17.04% -26.05%. The radial hardness decreased by 2.81% and 11.06% at 180°C treatments of 3 and 5 hours, respectively, while the duration of 7 hours caused an increase. Treatments of 200°C also reduced the radial hardness (2.68% - 18.86%). In each case of treatment, the impact bending strength of poplar wood was reduced (24.27%-62.02%), compared to control, while all treatments resulted in an increase of the compression strength of the samples (13.70% - 23.26%). Finally, only the less severe treatment managed to decrease slightly (2.80%) the roughness of wood surface, while increasing the temperature and duration of treatment, the degree of roughness sharply increased (26.92% to 70.36%).

In most of the cases, the effect of the temperature factor was found statistically significant and affected the variability of property values at a higher extent, compared to the treatment duration or the interaction between the two factors.

An ideal way of utilizing this modified species is to be used in applications where its new enhanced properties can be fully utilized, ensuring that the deteriorated properties will not be critical priority during the use. Wooden frames, floors, bathroom and kitchen structures, linings, decorative indoor/outdoor details, participation selectively in the construction of outdoor furniture under shelter or after additional protection, may be some possible applications of the specific materials.

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DURABILITY OF THERMO-HYDRO TREATED (THT) BIRCH VENEERS AND PLYWOOD

Ilze IRBE
Latvian State Institute of Wood Chemistry, Dzerbenes Str. 27, LV-1006, Riga, Latvia
Tel: +371 67553063, Fax: +371 67550635, E-mail: ilzeirbe@edi.lv

Juris GRININS
Latvian State Institute of Wood Chemistry, Dzerbenes Str. 27, LV-1006, Riga, Latvia
Tel: +371 67553063, Fax: +371 67550635, E-mail: jurisgrinins@inbox.lv

Bruno ANDERSONS
Latvian State Institute of Wood Chemistry, Dzerbenes Str. 27, LV-1006, Riga, Latvia
Tel: +371 67553063, Fax: +371 67550635, E-mail: bruno.andersons@edi.lv

Ingeborga ANDERSONE
Latvian State Institute of Wood Chemistry, Dzerbenes Str. 27, LV-1006, Riga, Latvia
Tel: +371 67553063, Fax: +371 67550635, E-mail: i.andersone@edi.lv

Abstract

The effect of thermo-hydro treatment (THT) on the durability of birch wood veneers and two plywood products – A and B was studied. THT was carried out in a multi-functional pilot scale wood modification device under elevated water vapour pressure at the following temperatures and treatment times (°C/min): 150/10; 150/50; 160/10 and 160/50. After THT, durability was examined according to the standards ENV 12038, EN 84 and EN 73. The durability class (DC) according to the CEN/TS 15083-1 was assigned to each treatment after fungal exposure. Water soluble extractives from THT samples were analysed with a UV-Vis Spectrometer in a range of 200 - 400nm, potentiometric titration and the Malaprade reaction.

The best DC (2 - 3) for aged veneers was reached with the THT 160°C against the white rot fungus. The THT of industrial plywood A at 160°C enabled producing a material with improved durability (DC 1 - 3) after ageing. Plywood B with glued THT veneers showed lower decay resistance than plywood A. After ageing, better performance of plywood B was achieved at the THT 160°C assigning to DC 2 - 4.

At the THT regimes under study, water soluble products (acids, sugars, aromatic compounds) were formed from both polysaccharides and lignin.

Key words: *Thermo-hydro treatment (THT); decay fungi; durability; veneer; plywood.*

INTRODUCTION

Plywood is an engineered wood-based product with improved physical and mechanical properties including high strength-to-weight and strength-to-thickness ratios, and excellent dimensional stability. Plywood panels are of great importance for furniture production and building construction and have been widely used for a variety of interior and exterior applications.

The properties of plywood depend on the quality of the different layers of veneer, the order of layer placement, the adhesive used, and the control of bonding conditions (Youngquist 1999). However, the application of plywood in exterior conditions is limited due to the sensitivity to moisture and biodegradation (Baileys et al. 2003).

Thermo-hydro treatment (THT) of wood at relatively high temperatures ranging from 150°C to 260°C is an alternative and environmentally friendly protection method in comparison to the chemical treatment with biocides. Temperatures around 160°C for hardwoods and around 180°C for softwoods are common (Militz 2002). An important effect achieved by the THT of wood is reduced hygroscopicity, and subsequently, increased dimensional stability (Biziks et al. 2015; Hillis 1984; Zaman et al. 2000) and improved durability (Boonstra et al. 2007; Irbe et al. 2014; Kamdem et al. 2002). Besides, the chemical, structural and mechanical properties of wood are changed (Andersons et al. 2016a; Biziks et al. 2016; Hill 2006).

There is limited information about the impact of THT on wood-based products. Several studies on plywood from thermally modified beech (Grzeskiewicz et al. 2009; Schulz et al. 2012), poplar

veneers (Zdravković et al. 2013) and the durability of different commercial wood-based products (Barnes et al. 2016) are reported. However, an industrial method has not yet been developed.

We have examined two approaches for producing THT plywood. The first is the THT of industrial plywood panels and the second is the THT of veneers prior to gluing to form a plywood panel. Thin veneers treated in an aggressive treatment environment (pressure, temperature and low pH values) react differently compared to solid wood. High treatment temperatures and exposure times, which are suitable for solid wood, are not suitable for veneers, because they become more brittle, resulting in a high loss of mechanical strength. Therefore, mild treatment parameters were chosen for veneers and plywood. Our investigations on different properties of THT veneers and two THT plywood products (A and B) have been carried out and partially published (Andersons et al. 2016b; Grinins et al. 2016a; Grinins et al. 2016b).

OBJECTIVE

In the present study, the durability of THT veneers and two THT plywood products – A and B were investigated according to the standards ENV 12038, EN 84 and EN 73. The thermal destruction products in water leachates were analysed and their effect on decay resistance was discussed.

MATERIAL, METHOD, EQUIPMENT

THT Procedure

THT plywood was obtained by two different methods. Plywood A represented THT industrial birch plywood glued with a phenol formaldehyde (PF) adhesive. Plywood B represented THT birch veneers glued with a PF film (temperature – 140°C, pressing time – 15 min, pressure – 1.2 MPa) to make plywood panels. The parameters of plywood A and B are given in Table 1.

Table 1

Parameters of plywood panels A and B

Plywood	THT applied	Panel dimensions [mm^3]	Plies [No]	Veneer thickness [mm]	Glue	Replicates [No]
A	Industrial panels	350 × 1000 × 18	13	1.4	PF adhesive	6
B	Veneer sheets	350 x 900 x 13	9	1.4	PF film, 220 g/m^2	6

THT was carried out in a pilot scale 540 L autoclave (WTT, Wood Treatment Technology) at elevated water vapour pressure, at four treatment regimes (max temperature, ˚C/ time, min): 150/10; 150/50; 160/10/ 160/50. The mild treatment regimes were chosen taking into account the sensitivity of birch solid wood against THT in our previous experiments, and the small thickness of veneers.

THT of the materials was carried out in three steps: heating, holding at the given temperature, and cooling. A calculated amount of water was pumped in at the beginning to generate saturated steam. Prior to the heating step, samples were held for 30min under a 0.02 MPa pressure in an autoclave for oxygen content reduction. The total time of treatment including the cooling down to room temperature depends on T_{max}, i.e. 24 - 25h (150°C) and 26 - 27h (160°C).

THT veneers and plywood panels were conditioned at a temperature of 20 ± 2°C and a 65 ± 5% relative humidity (RH) to equilibrium moisture content (MC).

Decay Test

The decay resistance was determined for (i) THT veneers with dimensions of 50 × 25 × 1.4mm^3, and (ii) THT plywood A and B with dimensions of 50 × 25 × thickness (mm^3) according to the modified European Prestandard ENV 12038:2002. Six specimens for each treatment were exposed to the brown rot fungus *Coniophora puteana* (BAM Ebw 15) and the white rot fungus *Trametes versicolor* (CTB 836A). Pine and birch wood specimens were used as virulence controls for *C. puteana* and *T. versicolor,* respectively. The fungi were cultivated on a medium containing 5% malt extract concentrate and 2% Fluka agar. In the THT veneer test, sterilised specimens were aseptically placed on 3mm steel supports in Petri dishes, and incubated at 22 ± 2°C and 70 ± 5% RH for 6 weeks. In the THT plywood test, the sterile specimens were placed on glass supports in Kolle flasks and cultivated for 16 weeks.

Subsequent to cultivation, the specimens were removed from the culture vessels, brushed free of mycelium and oven dried at 103 ± 2°C. The percentage mass loss (ML) of the specimens was the measure for the extent of fungal degradation.

Durability class (DC) according to the Technical Specification CEN/TS 15083-1:2005 was assigned to each treatment after fungal exposure: 1 – very durable (ML ≤ 5%); 2 – durable (ML > 5 to ≤ 10%); 3 – moderately durable (ML > 10 to ≤ 15%); 4 – slightly durable (ML > 15 to ≤ 30%); 5 – not durable (ML > 30%).

Ageing Procedures: Leaching and Evaporation

Prior to the ENV 12038 test, six THT veneers and plywood specimens from each treatment were leached with distilled water according to EN 84:1997. Additionally, the water extractive of the polymerised PF adhesive was obtained. UV absorption of diluted extracts was measured in a range of 200 - 400nm with UV-VIS Spectrometer Genesys™ 10. Acetic acid was potentiometrically titrated with 0.1M KOH solution using TIM 980 Titration Manager. The α-diol groups for sugar content analyses were determined *via* the reaction with periodate (Malaprade reaction) according to Meile et al. (2014).

Before the ENV 12038 test, six THT plywood specimens from each treatment were evaporated for 12 weeks in a wind channel according to EN 73:1988.

RESULTS AND DISCUSSION

Decay Resistance of THT Veneers

Decay test results for veneers are shown in Table 2. The ML of veneers (in both test procedures) is higher when subjected to *C. puteana;* possibly, because the virulence control shows a 43.4% ML vs. a 25.0% ML for *T. versicolor*. ENV 12038 results show that the lowest decay resistance against *C. puteana* is exhibited for veneers 150/10 (43%), whereas in the case of *T. versicolor,* the untreated and both 150/10 and 150/50 samples show a ML of 29%. T_{max} 160°C is most effective with this regard. After attack by *C. puteana,* the average ML of these veneers is 30%, but for *T. versicolor* 19%.

Table 2

Mass loss with SD and durability class (DC) of THT birch veneers after degradation by the decay fungi C. puteana and T. versicolor according to the test methods ENV 12038 and EN 84. Mean values, n = 6

Treatment temp./ time [°C/ min]	ENV 12038		ENV 12038 with EN 84	
	C. puteana	*T. versicolor*	*C. puteana*	*T. versicolor*
Untreated	36.6 ± 3.5 (DC 5)	29.6 ± 3.4 (DC 4)	30.6 ± 5.6 (DC 5)	9.7 ± 3.4 (DC 2)
150/10	43.0 ± 6.8 (DC 5)	29.3 ± 4.2 (DC 4)	31.3 ± 8.6 (DC 5)	18.4 ± 6.0 (DC 4)
150/50	38.6 ± 4.9 (DC 5)	28.8 ± 8.0 (DC 4)	40.7 ± 6.6 (DC 5)	18.8 ± 4.9 (DC 4)
160/10	29.1 ± 4.4 (DC 4)	19.6 ± 5.0 (DC 4)	27.9 ± 2.2 (DC 4)	12.1 ± 3.7 (DC 3)
160/50	30.7 ± 12.5 (DC 5)	18.1 ± 5.5 (DC 4)	18.8 ± 6.6 (DC 4)	6.7 ± 3.0 (DC 2)

In the combined ENV 12038/ EN 84 test, veneers 150/50 show the lowest decay resistance against *C. puteana* (40.7%), while both veneers 150/10 and 150/50 have the lowest decay resistance against *T. versicolor* (18%). Expectedly, the best ML results are obtained for veneers 160/50, namely 18.8% (*C. puteana*) and 6.7% (*T. versicolor*).

According to the CEN/TS 15083-1, the veneers treated at the given regimes are slightly durable to non-durable (DC 4 - 5) after the ENV 12038 test, and durable to non-durable (DC 2 - 5) after the leaching test (Table 2).

Our previous experiments with THT birch solid wood, treated at 160/3h and 170/1h, resulted in more durable samples against decay fungi (Irbe et al. 2014). The lower thickness (1.4mm) of the veneers tested in the present paper is one of the reasons for the differences, compared with the case of the experiments with solid wood samples (5mm thickness). Another reason could be the milder THT regimes tested in this study.

After leaching, water extractives were collected and analysed by UV-Vis spectrometry (Fig. 1): one band was around 202 - 206nm and another around 276 - 280nm. These bands are characteristic for aromatic compounds, and can be connected with the leaching of soluble lignin derivatives, polyphenol type compounds and/or furans, i.e. the destruction products of wood components (lignin, hemicelluloses). The extractives obtained from the veneers 160/50 have the highest absorptions, indicating that water soluble thermal degradation products are formed. The samples 160/10 have a lower absorbance but both samples 150/10 and 150/50 have the lowest values. Accordingly, serious degradation begins at 160°C. Extractives from the untreated sample do not display a band at 280nm and have a very low absorbance at 204nm.

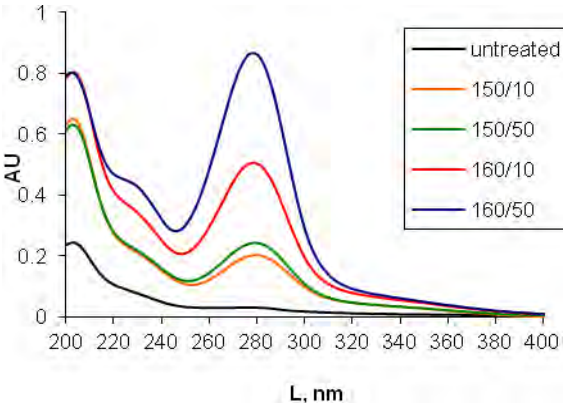

Fig. 1.
UV-Vis spectra of water extractives obtained from THT veneers.

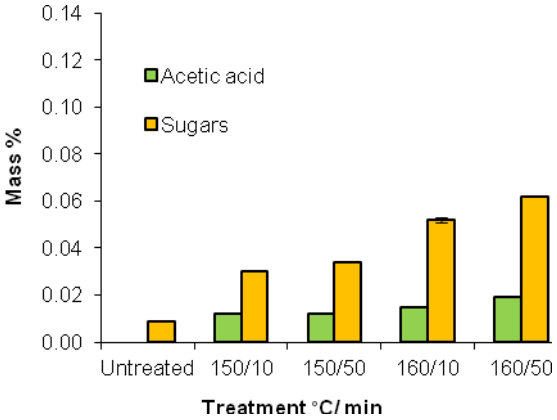

Fig. 2.
Total sugar (expressed as hexoses and pentoses) and acetic acid concentration in water extractives of veneers.

The origin of the UV bands is not always unambiguous as furfural and hydroxymethyl furfural and their condensation products are UV active, mainly at 280nm (Hon and Shiraishi 2000). Therefore, the more typical lignin band at 205nm is a better choice for lignin estimation, and this peak clearly shows that the THT time has no effect on lignin splitting, while a higher temperature increases the intensity of the 205nm peak.

Fig. 2 also demonstrates the efficiency increment from 150 to 160°C. However, the total amounts of water solubles are very small. These products are generated after hemicelluloses destruction – acetic acid is formed from acetyl groups, which split off during the thermal treatment and work as a catalyst for further depolymerisation of polysaccharides, generating monosaccharides (sugars) (Tjeerdsma et al. 1998). Our results partly explain the better decay resistance of veneers after leaching according to EN 84, i.e. the easily accessible products for enzymatic activity are removed by leaching.

The thermal degradation products are not toxic for both fungi as the ML after veneer leaching does not change much in the case of *C. puteana,* but for *T. versicolor,* ML even decreases probably because of leached nutrition compounds such as sugars etc. Untreated veneers demonstrate a similar trend, with a decreased decay rate after leaching.

Decay Resistance of THT Plywood A

The ML of THT plywood A in the test ENV 12038, and in the combined ENV 12038/ EN 73 test is higher when subjected to *T. versicolor*, but for the combined ENV 12038/ EN 84 test, it is higher for *C. puteana* (Table 3). In ENV 12038 and combined 12038/ EN 73 tests, both untreated and THT plywood A is very durable to durable (DC 1 - 2) to attack by fungi.

In the combined ENV 12038/ EN 84 procedure, the untreated plywood shows the lowest decay resistance against both fungi. The THT 150°C provides a low resistance against *C. puteana* (ML 26.3 - 31.7%) and *T. versicolor* (ML 18.0 - 21.3%). Better resistance against both fungi is achieved at the THT 160°C (DC 2 - 3). Despite this improvement, plywood A cannot be considered as very durable because the ML of the specimens is greater than 5% (8.4 - 14.5%).

Two bands from the UV spectra at 202 - 206nm and 276 - 280nm were analysed (Fig. 3). Extractives from the THT 160°C have the highest absorption for both bands, indicating that water soluble thermal destruction products are formed. Acid catalysed degradation of hemicelluloses leads to the formation of formaldehyde, furfurals and other aldehydes (Tjeerdsma et al. 1998). These hemicellulose destruction products more reliably show an absorption maximum at 280nm but the changes in the lignin structure are represented by the peak at 202 - 206nm. Although lignin is considered as the most thermally stable component of wood, its structure is changed by THT (Grinins et al. 2013).

Table 3

Mass loss with SD and durability class (DC) of THT plywood A after degradation by the decay fungi C. puteana and T. versicolor according to the test methods ENV 12038, EN 84 and EN 73. Mean values, n = 6

Treatment temp./ time [°C/ min]	ENV 12038		ENV 12038 with EN 84		ENV 12038 with EN 73	
	C. puteana	*T. versicolor*	*C. puteana*	*T. versicolor*	*C. puteana*	*T. versicolor*
Untreated	0.9 ± 0.3 (DC 1)	1.1 ± 0.3 (DC 1)	43.5 ± 1.6 (DC 5)	26.7 ± 1.1 (DC 4)	3.4 ± 0.4 (DC 1)	3.6 ± 0.2 (DC 1)
150/10	1.8 ± 0.7 (DC 1)	4.5 ± 3.9 (DC 1)	31.7 ± 4.8 (DC 5)	21.3 ± 1.5 (DC 4)	1.6 ± 0.3 (DC 1)	5.6 ± 7.2 (DC 2)
150/50	2.9 ± 0.8 (DC 1)	5.4 ± 3.7 (DC 2)	26.3 ± 5.5 (DC 4)	18.0 ± 2.0 (DC 4)	1.1 ± 0.2 (DC 1)	3.6 ± 0.5 (DC 1)
160/10	2.3 ± 0.3 (DC 1)	3.0 ± 1.1 (DC 1)	14.5 ± 2.6 (DC 3)	8.4 ± 0.8 (DC 2)	0.3 ± 0.3 (DC 1)	2.7 ± 0.2 (DC 1)
160/50	2.4 ± 0.6 (DC 1)	2.7 ± 1.1 (DC 1)	10.2 ± 4.7 (DC 3)	9.0 ± 1.0 (DC 2)	0.0 ± 0.3 (DC 1)	1.3 ± 0.1 (DC 1)

Untreated plywood shows no absorption at 280nm and low absorption at 204nm if compared with the THT sample peaks. Separate analyses of the water leachate of the polymerised PF adhesive demonstrated a higher UV absorption at 204nm than at 286nm (data not shown). Accordingly, the effect of the PF adhesive on the UV absorption spectra at 204nm cannot be excluded.

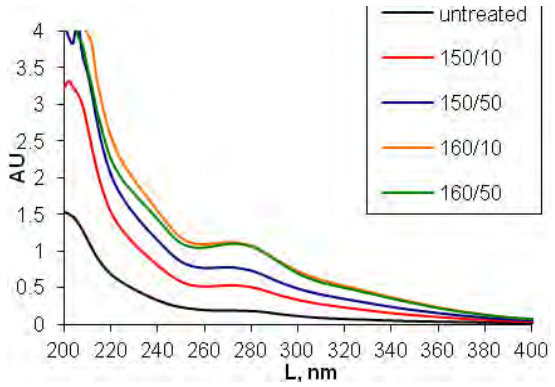

Fig. 3.
UV-Vis spectra of water extractives obtained from plywood A.

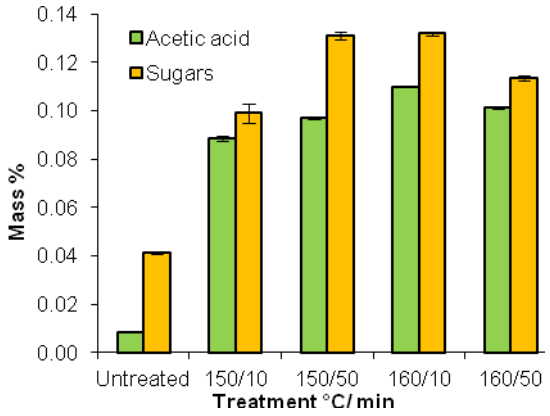

Fig. 4.
Total sugar (hexoses and pentoses) and acetic acid concentration in water extractives of plywood A.

The analyses of water extractives show the presence of some thermal destruction products (sugars and acid), with a tendency to increase with increasing THT regime (Fig. 4). These products are generated after hemicellulose destruction as described before. In plywood A, extracted sugars and acid amounts are higher than for THT veneers. At 160/50, the extracted amount slightly decreases, probably, because the THT time was long enough and decomposition continued, generating simple products like water and carbon dioxide or other easily volatile destruction products.

A significantly higher durability is observed for non-leached THT plywood A if compared with THT non-leached veneers (Table 2). Consequently, the fungal growth perhaps is influenced by the fungicidal effect of the PF adhesive (phenols, formaldehyde) rather than the THT destruction products. After leaching, the water soluble phenolic compounds from the adhesive are removed and plywood becomes susceptible to fungal attack similar to THT veneers.

Decay Resistance of THT Plywood B

The decay resistance of THT plywood B is similar to that of THT veneers (Table 4). The durability of plywood B is much lower than for plywood A after ENV 12038 and combined ENV 12038/ EN 73 tests.

The ML of THT plywood B after ENV 12038 ranges from 13.4% to 33.7% giving DC 3 - 5. In combined 12038/ EN 73, plywood B performs as a moderately durable to slightly durable (DC 3 - 4) material. In the combined ENV 12038/ EN 84, the THT 150°C ensures insufficient resistance against *C. puteana* (ML 29.2 - 32.8%) and *T. versicolor* (ML 17.9 - 27.4%). The best resistance against *T. versicolor* is achieved at the THT 160°C (DC 2 - 3). Nevertheless, plywood B cannot be considered as very durable because the ML is higher than 5% (7.9 - 11.3%). The low durability of plywood B could be attributed to the used PF film, which is made of PF resin impregnated paper. Apparently, the PF film is a more easily accessible substrate for fungal degradation than the PF adhesive in plywood A.

Table 4

Mass loss with SD and durability class (DC) of THT plywood B after degradation by the decay fungi C. puteana and T. versicolor according to the test methods ENV 12038, EN 84 and EN 73. Mean values, n = 6

Treatment temp./ time [°C/ min]	ENV 12038		ENV 12038 with EN 84		ENV 12038 with EN 73	
	C. puteana	*T. versicolor*	*C. puteana*	*T. versicolor*	*C. puteana*	*T. versicolor*
150/10	33.7 ± 4.8 (DC 5)	32.4 ± 2.2 (DC 5)	32.8 ± 2.0 (DC 5)	27.4 ± 4.7 (DC 4)	27.5 ± 6.0 (DC 4)	29.8 ± 3.9 (DC 4)
150/50	32.9 ± 5.0 (DC 5)	28.9 ± 1.4 (DC 4)	29.2 ± 2.2 (DC 4)	17.9 ± 5.9 (DC 4)	27.7 ± 5.6 (DC 4)	28.0 ± 2.7 (DC 4)
160/10	17.0 ± 3.6 (DC 4)	24.3 ± 1.8 (DC 4)	22.0 ± 1.4 (DC 4)	11.3 ± 1.2 (DC 3)	12.6 ± 4.9 (DC 3)	24.4 ± 1.2 (DC 4)
160/50	13.4 ± 1.8 (DC 3)	17.5 ± 1.3 (DC 4)	17.7 ± 3.5 (DC 4)	7.9 ± 1.6 (DC 2)	13.1 ± 5.8 (DC 3)	20.6 ± 2.3 (DC 4)

Similar to the THT veneers and plywood A, the UV bands at 202 - 206nm and 276 - 280nm of plywood B demonstrate the highest absorption for THT 160°C extractives, indicating that water soluble thermal destruction products are formed (Fig. 5). The THT 150°C has lower values.

The analyses of water extractives show the presence of thermal destruction products (sugars and acid), with a tendency to increase with increasing THT regime (Fig. 6). A pronounced amount of sugars is detected in leachates of THT 160°C samples. The mass percentage is three times higher than for THT 150°C samples and twice higher than at an equal regime for THT veneers. Perhaps, the gluing procedure of veneers into the plywood B plates at high temperature (140°C) and pressure (1.2 MPa) promotes some decomposition of structural oligomers to monomers.

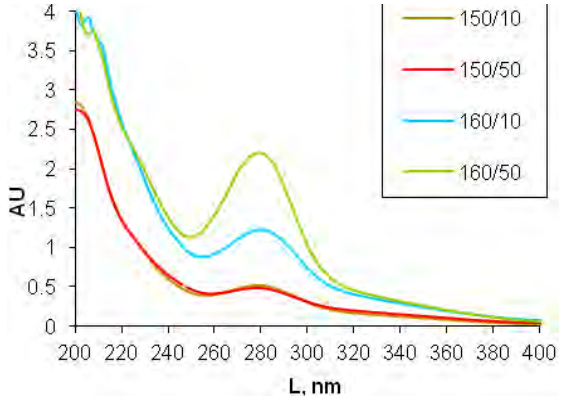

Fig. 5.
UV-VIS spectra of water extractives obtained from plywood B.

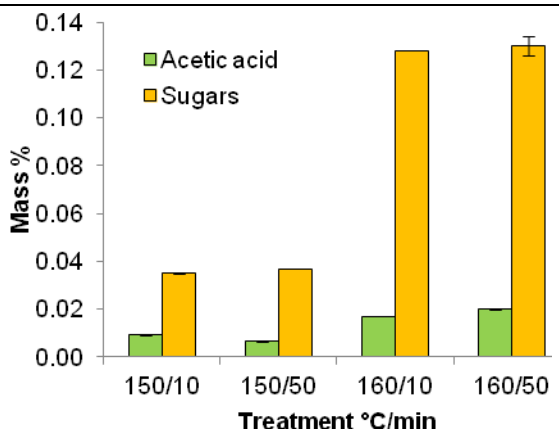

Fig. 6.
Total sugar (hexoses and pentoses) and acetic acid concentration in water extractives of plywood B.

The difference in durability rating between non-leached and leached THT plywood B (Table 4) is not as drastic as for plywood A (Table 3). This can be caused by the non-toxic behaviour of the PF film in comparison with the PF adhesive. Similar to THT veneers, the thermal destruction products have promoted the fungal degradation of plywood B samples (ENV 12038), while after the leaching, the decay capacity dropped.

CONCLUSIONS

The THT of birch veneers in comparatively mild conditions (150 and 160°C; 10 and 50min) does not allow obtaining of a very durable material (ML ≤ 5% according to CEN/TS 15083-1) against wood decay fungi. The best durability class (DC 2 - 3) of veneers is achieved by the THT 160°C against the white rot fungus *T. versicolor*. Water soluble compounds, produced at the given THT regimes, favour the decay ability of white and brown rot fungi because the ML declines after leaching.

The THT of birch plywood A at 160°C enables producing a material with improved resistance to decay fungi in outdoor exposure (moisture and evaporation) that is attributed to DC 1 - 3 (very durable to moderately durable). THT birch plywood B shows a lower decay resistance than plywood A. After the ageing procedures, the best performance of plywood B is achieved at the THT 160°C, attributed to DC 2 - 4 (durable to slightly durable). If compare two approaches of THT plywood producing, it is clear that the THT of veneers prior to the gluing with a PF film makes a product with a lower durability than the THT of industrial plywood which is glued with a PF adhesive.

At the THT regimes under study, substantial component changes occur in birch veneers and plywood, namely, water soluble extractives (acids, sugars, aromatic compounds) are formed from both polysaccharides and lignin.

Untreated birch plywood is very durable (DC 1) before leaching, probably because of the effect of the PF adhesive. After the non-polymerising adhesive components are leached out, the plywood becomes more accessible to fungal attack.

ACKNOWLEDGEMENT

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SELECTED PHYSICAL PROPERTIES OF THERMALLY MODIFIED SPRUCE WOOD

Adam SIKORA
Department of Wood Processing, Czech University of Life Sciences in Prague, Kamýcká 1176, Praha 6 - Suchdol, 16521 Czech Republic
Tel: +420 22438 3737, Fax: +420 22438 3737, E-mail: sikoraa@fld.czu.cz

Štěpán HÝSEK
Department of Wood Processing, Czech University of Life Sciences in Prague, Kamýcká 1176, Praha 6 - Suchdol, 16521 Czech Republic
Tel: +420 22438 3737, Fax: +420 22438 3737, E-mail: hyseks@fld.czu.cz

Milan GAFF
Department of Wood Processing, Czech University of Life Sciences in Prague, Kamýcká 1176, Praha 6 - Suchdol, 16521 Czech Republic
Tel: +420 22438 3737, Fax: +420 22438 3737, E-mail: gaff@fld.czu.cz

Petr HORAČEK
Department of Wood Processing, Czech University of Life Sciences in Prague, Kamýcká 1176, Praha 6 - Suchdol, 16521 Czech Republic
Tel: +420 22438 3737, Fax: +420 22438 3737, E-mail: petrtomashoracek@seznam.cz

Veronika VONDROVA
Department of Wood Processing, Czech University of Life Sciences in Prague, Kamýcká 1176, Praha 6 - Suchdol, 16521 Czech Republic
Tel: +420 22438 3737, Fax: +420 22438 3737, E-mail: veronika.vondrova@seznam.cz

Abstract

Thermal modification of wood does not only change the color of the wood, it also changes wood properties that are closely related to its reaction to water (changes in dimensions, weight, density,...). These properties of thermally modified wood play an important role, limiting the use of thermally modified wood itself. These changes may be positive or negative depending on the wood species, thermal modification mode, the temperature used during the modification and other factors.

The aim of this article is to broaden and collect knowledge about the effect of different thermal modification temperatures (160°C, 180°C and 210°C) on selected physical characteristics of spruce wood.

The results of the study show that thermal modification at a temperature range of 160°C – 210°C has a statistically significant effect on the absorbability of thermally modified wood, as well as on tangential and volumetric swelling. Conversely, the effect of thermal modification on longitudinal swelling and a change in density during absorption was not proven.

Key words: *absorption; density; thermally modified wood.*

INTRODUCTION

Modifying wood with high temperatures is currently considered a suitable way to regulate the resulting physical properties of wood. Thermal modification eliminates the application of toxic agents necessary to improve wood resistance, and it improves dimensional stability, thereby reducing the hygroscopic behavior of the material, but the density of the wood also decreases. High-temperature treatment of material can be considered a method of wood modification in which the wood's color can be modified (Romagnoli et al. 2007).

In terms of the practical use of thermally modified wood, its resulting physical properties are considered to be an important factor (Barcík 2015), and the color as such can be an indicator of the quality of the thermally modified wood, determining its final use in the market (Boonstra 2008). During thermal modification, the wood darkens and acquires a different shade. In the past, the thermal modification process has been applied to remove unwanted color differences between the sapwood and the heartwood, or to remove stains that form during wood steaming (Tolvaj and Faix 1996). The

color characteristics depend mostly on the specific chemical composition of the wood in interaction with light (Hon and Shiraishi 2001). Dry spruce wood is chemically composed of cellulose (40-50%), hemicelluloses (25-30%), lignin (25-30%), and extractives (3-10%). These proportions are only approximate, and they are influenced by multiple factors (Fengel and Wegener 2003). Initial changes in physical properties due to thermal modification begin to manifest themselves at 150 °C (Gunduz et al. 2008). Changes in wood properties are primarily due to the degradation of hemicelluloses and the production of water, carbon dioxide, formic acid, acetic acid, and other substances that may be involved in condensation reactions, thereby forming chromophore groups (Hakkou et al. 2005). These chemical reactions resulting from high temperatures significantly reduce tangential and radial swelling (Gunduz et al. 2009), reduce the moisture content and water absorption of the wood (Kortelainen 2011), and also affect the fire resistance of the spruce wood (Čekovská et al. 2017).

Significant changes in all physical properties of thermally modified wood are observed at temperatures around 180 °C – 250 °C (Patzelt et al. 2003). At these high temperatures, a high dimensional stability of 55 - 90% was found. In spruce wood, a weight loss of 16.1% was found at a temperature of 210 °C (Gunduz et al. 2009). The change in color, moisture absorption, weight and dimensions is not only affected by the applied temperature, it is also greatly influenced by the period over which the wood is subjected to the temperature (Patzelt et al. 2003). At 250 °C, the process of wood carbonization begins, resulting in the formation of carbon oxides along with other substances (Kačíková et al. 2008).

OBJECTIVE

The objective of the study was to determine the effect of the thermal modification temperature the weight, volume, density and dimensional changes during absorption at various thermal modification temperatures.

MATERIAL, METHOD, EQUIPMENT

Spruce test specimens (Picea abies L.) were used for the research. The wood came from the Poľana region in Slovakia. The research consisted of two sets of test specimens:

- A set of test specimens not subjected to thermal modification (S-20 °C).
- A set of thermally modified test specimens (S-160 °C, S-180 °C, S-210 °C).

The following effects were monitored:

- The effect of the thermal modification on dimensional and volumetric swelling, absorbability and density of the wood during the absorption process.

To assess the swelling, we used 20 x 20 x 30 (h x w x l) test specimens (Fig.1).

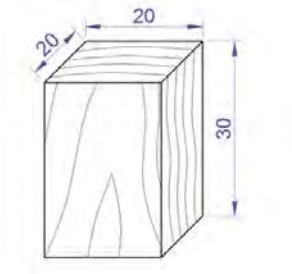

Fig. 1.
Test specimens for swelling evaluation.

The thermal modification process was carried out according to the following steps:

1. Heating and drying – In this stage, the temperature increases rapidly in an oven at about 100 °C to support the action of the steam. Then, the pitch decreases and increases to a level of 130 °C. The drying medium is hot air or hot steam. Throughout this phase, the wood is dried to approximately zero moisture content.

2. Thermal modification – In the second stage, the temperature is raised to 185-230 °C for 2-3 hours. The height of the temperature and duration of action are determined by the requirements for the class of THERMOWOOD products (Thermo-S and Thermo-D)

3. Refrigeration and air conditioning – In the third phase, the thermally modified wood is gradually cooled to a temperature of 80-90 °C, and the humidity is stabilized so that the final moisture level is in a normal range of 4 - 7%.

The parameters of the thermal modification of the test specimens are shown in Table 1, and the thermal modification mode is shown in Figure 2.

Table 1

Input technological parameters and thermal modification process

	Input technological parameters		
Wood moisture content	2 to 4 %		
Filled kiln capacity	0.8 m^3		
Maximum temperature achieved	210 °C		
	Thermal modification process		
	160 ° C	**180° C**	**210° C**
Heating	6.3 Hours	4.5 Hours	4.8 Hours
Thermisation	4.4 Hours	5.3 Hours	6.7 Hours
Cooling	1.7 Hours	2.4 Hours	4.2 Hours
Total modification time	**12.4 Hours**	**12.2 Hours**	**15.7 Hours**

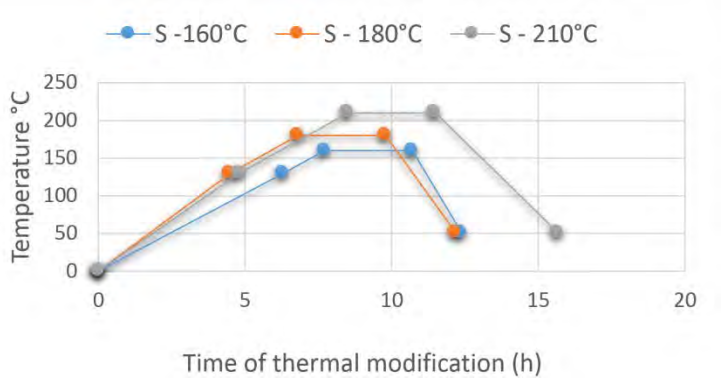

Fig. 2.
The thermal modification process specimens (S-210°C, S-180°C, S-160°C).

Methods

Absorption

The absorption indicators were values of selected characteristics measured on test specimens subjected to thermal modification. These measured values were compared with the values measured on test specimens that were not subjected to thermal modification. The following characteristics were monitored:

- Dimensional gain in individual wood directions (tangential, radial and longitudinal).
- Volumetric gain
- Weight gain
- Density gain over time, from dry state to full water saturation of the wood.
- Wood moisture content over time, from absolute dry state to full water saturation of the wood.

We determined the increase in individual monitored characteristics by measuring them at certain time intervals (0hr., 0.06hr., 0.2hr., 1hr., 3hrs., 6hrs., 12hrs., 24hrs., 48hrs., 72hrs., 96hrs.). The density of the measured materials is higher at the beginning of the measurement due to the faster reaction of the material to the moisture change (Fig. 4).

Evaluation and Calculation

Increase in monitored characteristics

The total and partial swelling of the wood over time was carried out in accordance with **ČSN 49 0104:** 1988. In order to determine the wood swelling values, the specimens were submerged in buckets filled with water, weighed down and left for 96 hours.

$$\beta_t = \frac{\beta_o - \beta_{wt}}{\beta_o} (\%) \quad (1)$$

Where β_t indicates the gain percentage in each direction depending on the monitored time, β_o is the dimension of the specimen in absolutely dry state, and β_{wt} specifies the dimensions of specimens in individual directions after each interval has elapsed.

We recorded the weight and dimensions in each direction (radial, tangential and longitudinal) at the given time intervals. We then used the measured data to calculate the increase in the monitored value over time Eq. 2,

For the completeness of the values needed to calculate the swelling over time and the overall swelling, the results from the density measurements at 0% moisture content were used. Eq. X.

$$P_1 = Ch_{t2} - Ch_{t1} \quad (2)$$
$$P_2 = Ch_{t3} - Ch_{t1}$$
$$P_3 = Ch_{t4} - Ch_{t1}$$

where $P_{(1,2,3,.....)}$ is the increase in the monitored characteristic over time t_1, t_2, t_3, $Ch_{t(1, 2, 3, ...)}$ is the value of the monitored characteristic over time t_1, t_2, t_3, and Ch_{t1} is the value of the monitored characteristic at the beginning of the measurement, i.e. at t_1. The increments were measured until the limit of hygroscopicity was reached - the increments were measured at predetermined intervals for 96 hours.

We used this method to measure and calculate:

1. The weight gain (g) after 96 hours of soaking.
2. The volumetric gain (mm3) after 96 hours of soaking.
3. The density gain (kg.m3) after 96 hours of soaking.
4. The radial gain (mm) after 96 hours of soaking.
5. The tangential gain (mm) after 96 hours of soaking.
6. The longitudinal gain (mm) after 96 hours of soaking.

ANOVA (Fisher's F – test) and Duncan's multiple comparison tests were used to evaluate the statistical significance of individual factors. A 0.05% level of significance was used for all statistical analyses. Statistical analyses were performed using STATISTICA 12 (Statsoft Inc., USA).

The wood density was determined according to ISO 13061-2 (2014) after drying and after 96 hours. Eq. 3,

$$\rho_w = \frac{m_w}{a_w * b_w * l_w} = \frac{m_w}{V_w}, \quad (3)$$

where ρ_w is the sample density at a certain moisture content w (kg/m^3), m_w is the sample mass at a certain moisture content, w (kg), a_w, b_w, and l_w are the sample dimensions at a certain moisture content w (m), and V_w is the sample volume at a certain moisture content w (m^3).

The moisture content in the samples was determined according to ISO 13061-1 (2014) and Eq. 4,

$$w = \frac{m_w - m_0}{m_0} * 100 \quad (4)$$

where w is the moisture content of the sample (%), m_w is the sample mass at a certain moisture content w (kg), and m_0 is the sample mass in a dry state (kg).

RESULTS AND DISCUSSION

Increase in monitored characteristics

Table 2 shows the average values of increments in the monitored characteristics over time, from dry state to their complete saturation with water.

Table 2

Mean values of increments in monitored characteristics after 96 hours

Wood Species	Thermal treatment (°C)	Weight gain (g)	Volumetric gain (mm^3)	Density gain (kg.m3)	Radial gain (mm)	Tangential gain (mm)	Longitudinal gain (mm)	Density (Kg/m^3)
Spruce	**20**	5.6 (7,1)	2208.1 (4.5)	326 (10.3)	2.1 (7.2)	3.3 (4.5)	1.1 (8.0)	445 (5.39)
Spruce	**160**	6.1 (18,7)	2515.7 (9.6)	336 (21.6)	2.2 (6.4)	3.4 (4.3)	1.4 (3.3)	449 (4.45)
Spruce	**180**	4.8 (12,9)	1956.2 (12.8)	313 (22.0)	1.9 (11.6)	2.9 (4.5)	1.2 (2.8)	467 (1.96)
Spruce	**210**	4.3 (12,2)	1663.3 (14.6)	268 (11.2)	1.9 (16.8)	2.5 (5.6)	1.1 (3.6)	461 (4.94)

Values in parentheses are coefficients of variation (CV) in %

Based on the significance level "p", we can say that a statistically significant effect of the thermal modification can be observed in the following characteristics: weight gain, volumetric gain, radial and tangential dimensional gain (Tab. 3). The density gain and the longitudinal dimensional gain are characteristics on which the thermal modification has no statistically significant effect.

Table 3

Statistical evaluation of the effect of thermal modification on the increase in monitored characteristics

Weight gain (g) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	1088.058	1	1088.058	2050.755	***
Temperature of Thermal treatment (TT)	19.468	3	6.489	12.231	***
Error	19.100	36	0.531		
Volumetric gain (mm^3) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	174030679	1	174030679	3668.397	***
Temperature of Thermal treatment (TT)	3951034	3	1317011	27.761	***
Error	1707859	36	47441		
Density gain ($kg.m^3$) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	3866491	1	3866491	890.6155	***
Temperature of Thermal treatment (TT)	27580	3	9193	2.1176	NS
Error	156289	36	4341		
Radial gain (mm) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	164.187	1	164.187	3451.443	***
Temperature of Thermal treatment (TT)	0.798	3	0.266	5.590	***
Error	1.713	36	0.048		
Tangential gain (mm) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	370.090	1	370.090	18214.322	***
Temperature of Thermal treatment (TT)	5.019	3	1.673	82.340	***
Error	0.731	36	0.020		

Longitudinal gain (mm) after 96 hours					
Evaluated Factor	**Sum of Squares**	**Degree of Freedom**	**Variance**	**Fisher's F-test**	**Significance Level p**
Intercept	57.701	1	57.701	1115.184	***
Temperature of Thermal treatment (TT)	0.170	3	0.057	1.098	NS
Error	1.863	36	0.052		

NS- not significant, ***- significant, Significance was accepted at P < 0.01

For the purpose of a deeper analysis of the effect of thermal modification on the monitored characteristics, we used Duncan's multiple comparison test; the results are shown in Table 4.

It is clear from the results that no statistically significant difference was found between the values of weight gain measured in the set of specimens modified at 160°C and the set of test specimens not subjected to thermal modification (20°C) (P = 0.172). A statistically insignificant difference (P = 0.170) in the measured weight gain values was also found between the values measured in sets of test specimens subjected to thermal modification at 180°C and 210°C.

The analysis of the effect of the thermal modification on volumetric gain values showed a statistically significant difference between all the monitored degrees of thermal modification (Tab. 4).

The results of the one-factor analysis of variance evaluating the effect of the thermal modification on the density gain listed in Table 3 show that the thermal modification does not have a statistically significant effect on the values of this characteristic. The results of Duncan's test shown in Table 4 indicate that there is a statistically significant difference between sets of test specimens modified at 160°C and 210°C, with a significance level of p = 0.038.

The results of Duncan's test show that there is no statistically significant difference between the radial gain values measured in sets of test specimens modified at 160°C, 180°C and without thermal modification (20). A statistically significant difference was found between the values of untreated specimens and sets of test specimens modified at 210°C (p = 0.014). A significant difference was also confirmed between the values measured in different sets of test specimens (160 °C – 180°C, p = 0.011; 160°C – 210°C, p = 0.002).

Thermal modification has a statistically very significant effect on the tangential gain; a statistically significant difference between all the sets of test specimens was found, with a significance level of p = 0.02 or less.

The longitudinal gain was not affected by the thermal modification in any of the monitored cases of thermal modification.

Table 4

Comparison of the effects of individual factors on the values of the gain of monitored characteristics using Duncan's test

Weight gain (g) after 96 hours					
Thermal treatment (°C)		**(1) 5.6480**	**(2) 6.1020**	**(3) 4.7840**	**(4) 4.3280**
S	**20**		0.172	0.012	0.000
S	**160**	0.172		0.000	0.000
S	**180**	0.012	0.000		0.170
S	**210**	0.000	0.000	0.170	
Volumetric gain (mm^3) after 96 hours					
Thermal treatment (°C)		**(1) 2208.1**	**(2) 2515.7**	**(3) 1956.2**	**(4) 1663.3**
S	**20**		0.003	0.014	0.000
S	**160**	0.003		0.000	0.000
S	**180**	0.014	0.000		0.005
S	**210**	0.000	0.000	0.005	
Density gain ($kg.m^3$) after 96 hours					
Thermal treatment (°C)		**(1) 326.44**	**(2) 336.47**	**(3) 312.91**	**(4) 267.79**
S	**20**		0.736	0.649	0.067
S	**160**	0.736		0.458	0.038
S	**180**	0.649	0.458		0.135
S	**210**	0.067	0.038	0.135	

Radial gain (mm) after 96 hours					
Thermal treatment (°C)		**(1) 2.1150**	**(2) 2.2060**	**(3) 1.9320**	**(4) 1.8510**
S	**20**		0.357	0.069	0.014
S	**160**	0.357		0.011	0.002
S	**180**	0.069	0.011		0.412
S	**210**	0.014	0.002	0.412	
Tangential gain (mm) after 96 hours					
Thermal treatment (°C)		**(1) 3.2770**	**(2) 3.4330**	**(3) 2.9490**	**(4) 2.5080**
S	**20**		0.020	0.000	0.000
S	**160**	0.020		0.000	0.000
S	**180**	0.000	0.000		0.000
S	**210**	0.000	0.000	0.000	
Longitudinal gain (mm) after 96 hours					
Thermal treatment (°C)		**(1) 1.1010**	**(2) 1.2830**	**(3) 1.2190**	**(4) 1.2012**
S	**20**		0.110	0.282	0.331
S	**160**	0.110		0.533	0.455
S	**180**	0.282	0.533		0.862
S	**210**	0.331	0.455	0.862	

The weight gain of test specimens with different thermal treatment is shown in Fig. 3. The graph clearly shows that there was a slight statistically insignificant increase in values by 9%, due to the effect of thermal treatment at 160°C, compared to the values measured in the set of specimens that were not subjected to thermal modification. In contrast, Li et al. (2011) achieved a slight increase in the weight gain of wood thermally modified at 180°C, which was also statistically insignificant. Thermal modification at 180°C reduced the weight gain by 14.3% in comparison with untreated test specimens, and in specimens thermally modified at 210°C the weight gain values decreased by up to 23.3%. Increasing temperatures during thermal modification leads to a decrease in the water absorption capacity of spruce wood (Metsä-Kortelainen et al. (2006)). The lowest weight values at temperatures higher than 200°C have been achieved by many authors (Li *et al.* (2011), Korkut and Guller (2008), Chaouch *et al.* (2010) a Brito *et al.* (2008)).

The volumetric gain values (Fig. 4) measured in the monitored sets of test specimens have a similar course to that of the weight gain (Fig. 12). A statistically significant difference between all the sets of test specimens was confirmed. In comparison with the set of untreated test specimens (20°C), we found a 13.7% increase in the values of the monitored characteristic due to the application of 160°C. A decrease of 13.6% was caused by the application of 180°C, and a decrease of up to 22% was caused by the application of 210°C. Dimensional stability is one of the most important properties of wood materials, especially when they are exposed to high-performance conditions. A statistically significant difference in the volumetric gain of thermally modified wood was confirmed by Guller (2012).

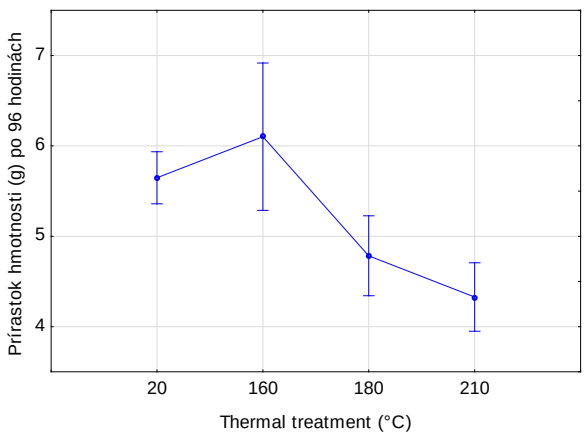

Fig. 3
The effect of thermal modification on the weight gain during soaking

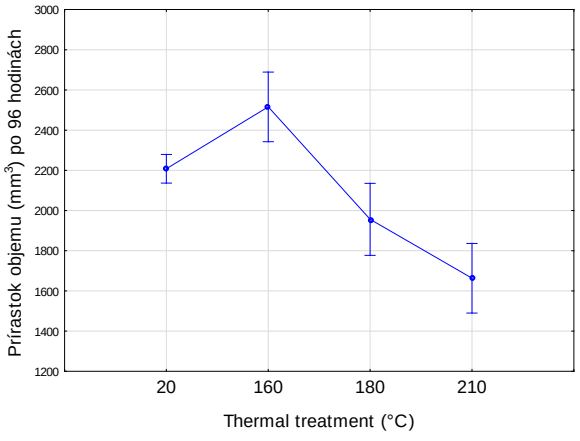

Fig. 4
The effect of thermal modification on the volumetric gain during soaking

The results showing the effect of the thermal modification on the density gain are listed in Figure 5. The values in the graph confirm the results listed in Table 9, as well as the results of Duncan's test (Tab. 4), on the basis of which we can conclude that the density gain is not affected by the thermal modification. Wood density significantly affects the properties of wood (Tsoumis 1991). Other monitored properties shown in Figures 6, 7 and 8 also have a similar density trend. Guller (2012), Kamdem et al. (2002)) and Chaouch et al. (2010) reported a decrease in wood density values with the application of temperatures higher than 200 °C.

Figure 6 shows a decrease in the radial gain values, which is, however, not significant between untreated sets of specimens (20 °C) and specimens thermally modified at 160 °C, 180 °C, based on Duncan's test results. A significant difference was confirmed between 20 °C and 210 °C sets of specimens. Thermal modification of wood significantly affects the radial swelling of wood. This effect was confirmed by Korkut and Guller (2008), Gündüz et al. (2008)

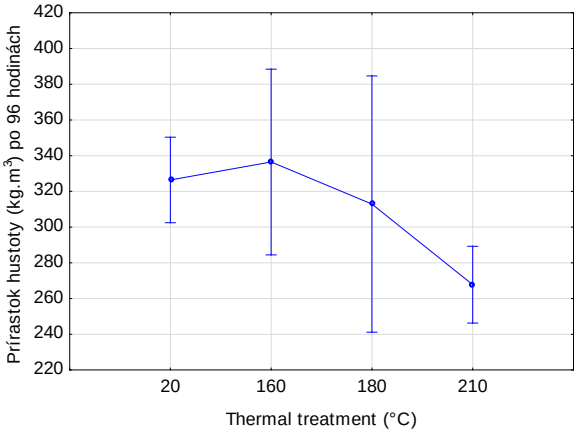

Fig. 5
The effect of thermal modification on the density gain during soaking

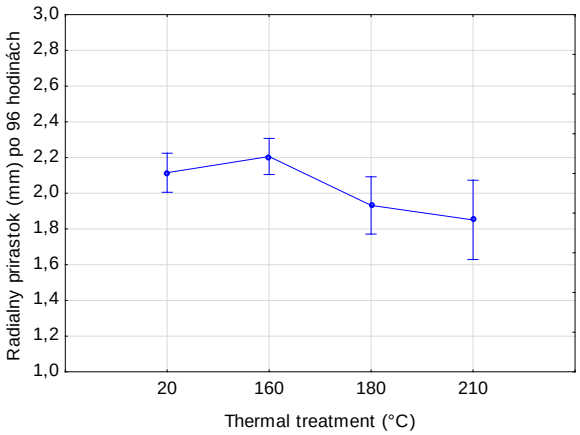

Fig. 6
The effect of thermal modification on the radial gain during soaking

Among the monitored characteristics, the tangential gain is most affected by the thermal modification (Fig. 7), which is evident from the results of the one-factor analysis of variance (Tab. 3), as well as the results of Duncan's test (Tab. 4). From a comparison of the tangential swelling and the radial swelling, it is evident that higher swelling values were achieved in the tangential direction. Higher tangential values were achieved by Gündüz et al. (2008). It was also found that as the temperature increases, with the exception of 180 °C, the swelling in the tangential direction decreases (Korkut and Guller (2008)).

The thermal modification temperature does not affect the values of the longitudinal gain during soaking (Fig. 8). Slight differences in swelling values in the longitudinal direction at different temperatures were also achieved by Gunduz *et al.* (2009).

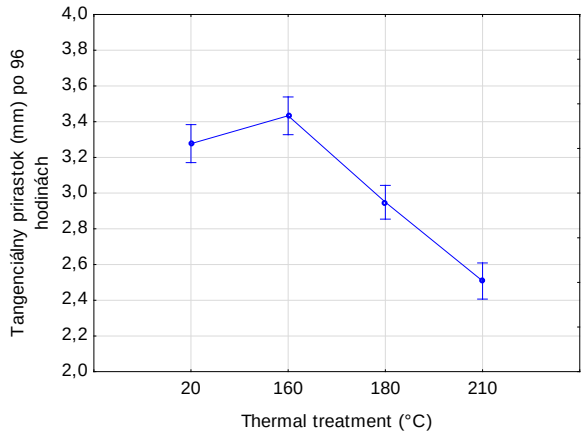

Fig. 7
The effect of thermal modification on the tangential gain during soaking

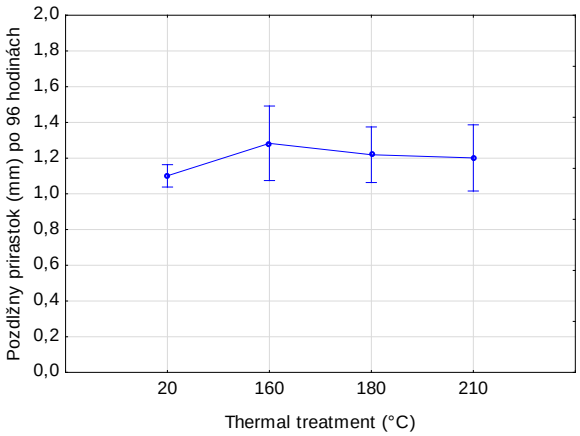

Fig. 8
The effect of thermal modification on the longitudinal gain during soaking

Fig. 9 shows the effect of thermal modification on the moisture absorption of wood. The graph shows a clear declining trend in the water absorption in correlation with the increasing temperature of the modification. The highest water absorption values were found in untreated wood, and the lowest moisture content was measured in a set of spruce specimens treated at 210 °C. A decrease in water absorption with the increasing temperature of thermally modified wood was also confirmed by Li *et al.* (2011) and Metsä-Kortelainen et al. (2006).

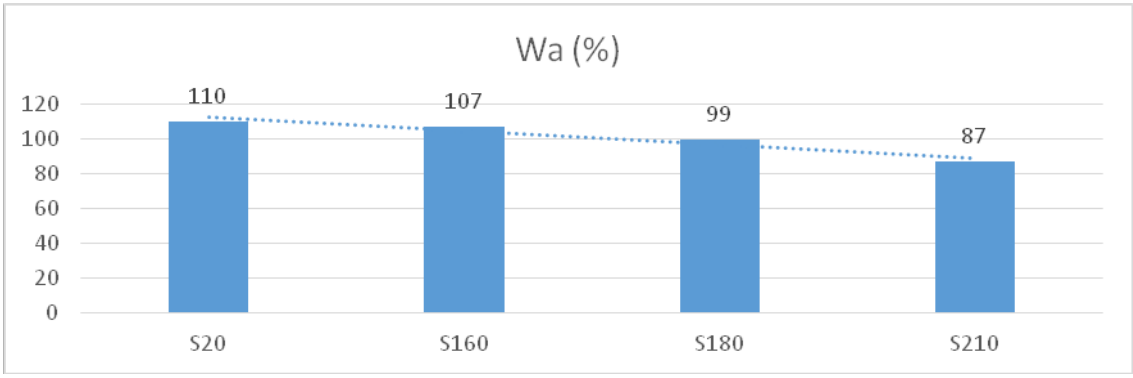

Fig. 9.
The effect of thermal modification on the water absorption

CONCLUSIONS

1. Different thermal modification temperatures have a statistically significant effect on radial and tangential swelling, but they do not have a statistically significant effect on longitudinal swelling. The effect of thermal modification on volumetric swelling was also confirmed.
2. The effect of the thermal modification on the wood density during the soaking process was not confirmed.

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EFFECTIVE SPECIFIC HEAT OF WOOD BRIQUETTES

Daniela SOVA
Transilvania University of Brasov, Faculty of Mechanical Engineering
Str. Politehnicii nr. 1, 500024 Brasov, Romania
E-mail: sova.d@unitbv.ro

Abstract

The effective specific heat of wood briquettes was determined by using a mathematical model developed for bulk pellets/briquettes and by measurements made in parallel and perpendicularly to the briquettes axis in the range of moisture contents below and above equilibrium moisture content. The mathematical model was based on the assumption that bulk pellets/briquettes are a porous system, consisting of solid particles and gas, with effective thermal properties. The model applied to the briquettes with the moisture content above the equilibrium moisture content is different to that applied to the briquettes with the moisture content below equilibrium moisture content, since alongside with wood fiber swelling, the magnitude and the number in the voids between chips increases. That is why, the briquettes density decreased in this moisture content range. New equations are proposed for this moisture content range considering a larger wood cell lumen that includes the chips interspaces. Experiments were performed at the temperature of briquettes ranging from 19 to 24°C and the average moisture content from 0 to 22.7%, dry basis. When increasing the moisture content above equilibrium moisture content, the briquettes expanded due to fiber swelling, but also due to the increase in the magnitude and number of chips interspaces. Both swelling and increase in briquettes voids influenced the effective specific heat.

Key words: *wood briquettes; effective specific heat; moisture content; transient line heat source method.*

INTRODUCTION

Romania is estimated to have a biomass energy potential of 7,594,000 toe/year corresponding to 19% of the total average primary consumption, which is identified in firewood and wood waste from harvesting operations (1,175,000 toe/year), sawdust and wood waste from wood processing operations (487,000 toe/year), agricultural waste (4,799,000 toe/year), biogas (588,000 toe/year) and household waste (545,000 toe/year) (Borz et al. 2013). Wood-processing residues (fine waste), such as sawdust and wood shavings, are considered to be of little economic value. They are either burned on site or transported to disposal sites. Compressing the low density biomass into a solid fuel of a convenient size and shape allows burning them in the same way like wood or charcoal. In this situation, briquettes could offer a means of waste management (Chaney 2010).

Briquettes are manufactured by compression of residues with a low moisture content (<25%, dry basis) at moderate to high pressure (>5MPa). Briquetting increases the bulk density of the biomass, increasing its energy density (the energy content per unit volume of material) (Chaney 2010).

Biomass fuel properties for the combustion analysis are grouped into physical, chemical, thermal and mineral properties. The thermal properties are specific heat, thermal conductivity and emissivity that vary with moisture content, temperature and degree of thermal degradation (Saidur et al. 2011, Ragland et al. 1991). Knowing the thermal properties of biomass briquettes and pellets is important for modeling the combustion process. Effective thermal conductivity and specific heat of bulk wood pellets are also important properties for studying self-heating during their storage (Guo et al. 2012). A packed bed of pellets is assumed by Guo et al. (2012) and Guo (2013) to be a continuous homogeneous porous system with effective thermal properties. Sjöström and Blomqvist (2014) used the transient plane source technique to measure the specific heat and thermal conductivity of bulk wood pellets within a temperature range of 22 and 120°C. They also investigated the possibility of measuring those properties on individual pellets while studying the moisture content dependence. The effect of moisture content on thermal properties of alfalfa pellets was studied by Fasina and Sokhansanj (1995) using the line heat source method for pellets moisture content ranging from 7.5 to 18%, wet basis. Specific heat and thermal conductivity of softwood, softwood bark and softwood char were comparatively measured at temperatures between 40 and 140°C by Gupta et al. [9].

As compared to wood, which is an anisotropic and heterogeneous biological porous material, briquettes are considered to be isotropic because of the random orientation of fibers during the

briquetting process. There is less information on specific heat of a single wood briquette and its dependence on moisture content and no information about its values if it is measured in parallel or perpendicularly to the briquette axis.

OBJECTIVE

The objectives of the research reported below were to investigate the specific heat of wood briquettes by measuring it using the transient line heat source method and by modeling it using the mathematical model developed for the effective specific heat of bulk pellets/briquettes. Both, measurement and modeling were carried out for moisture contents ranging from 0% to equilibrium moisture content and from equilibrium moisture content up to the maximum moisture content (24.5%, dry basis) permissible for briquettes to maintain their shape. Also, specific heat measurements were performed in parallel and perpendicularly to the briquettes axis.

MATERIAL, METHOD, EQUIPMENT

The briquettes were formed by compression of wood processing residue, i.e. softwood and hardwood chips in uncontrolled amounts, in a hydraulic briquetting press (MB4 Goldmark). Compression is a continuous extrusion process which depends on the friction forces from the side of the die acting to produce compression. The pressure used to form cylindrical briquettes with densities between 750 and 800kgm^{-3} was 150bar. The resulting dimensions of the briquettes were 40mm for the diameter and 30-75mm for the length. The maximum moisture content required by the producer of the briquetting press is 17%. No binders were used in forming the briquettes.

Twenty briquettes were selected for thermal conductivity and specific heat measurements from a lot of 200 extruded briquettes. They were stored in the Laboratory of Heat Transfer at 20±1^{o}C and 45±2%RH. Two lengths and two diameters of each briquette were measured using a digital pocket caliper (ULTRA, 0.01 mm accuracy). A stereometric method (Rabier et al. 2006) was used to determine briquette density. This method was chosen and not a displacement method, in order to preserve the structure of the briquettes which otherwise could have alter the measurement of thermal properties. The stereometric method consisted in briquettes weighing using a mass balance (KERN-EW 3000g, 0.01g accuracy), calculating the volume of the briquettes by using their main dimensions and determining the density.

The briquettes were afterwards oven dried at 103±2^{o}C to constant mass in order to determine the moisture content (dry basis). In order to prevent loss of material during briquettes handling, drying and weighing, previously dried glass pans were used. The moisture content was calculated based on wet and dry briquette masses (SR EN 13183-1-2003/AC-2004). The briquettes dimensions were measured again after oven drying and briquettes density was recalculated.

Thermal conductivity and volumetric specific heat were measured with KD2 Pro analyzer (Decagon Devices Inc.) by using a SH-1 dual-needle sensor (30mm length, 1.30mm diameter, 2 needles, 6mm spacing), based on the transient line heat source method (Chaney 2010, Speyer 1996). This method consists in the generation of a heat pulse by one probe, the response measured by the other, and a numerical analysis of the response behavior, which allows the thermal properties (volumetric heat capacity, thermal conductivity and thermal diffusivity) to be found. In order to apply the method, two Ø=1.3mm x 30mm orifices were drilled in each briquette. From the amount of twenty briquettes, nine were drilled in parallel with the briquettes axis and the other nine perpendicularly to the briquette axis. The last two briquettes were drilled both parallel and perpendicularly to the axis. Two or three measurements of the thermal properties were performed for each briquette, at 0% moisture content (*MC*) and equilibrium moisture content (*EMC*).

Thermal properties of briquettes depend on many factors, such as the material from which they are made, the density to which they are compressed and the moisture content. In the case of biomass briquettes, due to their varied nature in terms of constituent materials, the conditions under which they are formed and their moisture content, standard literature values are not available; there are likely to be significant differences between the thermal properties, not only for briquettes of different materials, but also for briquettes of the same material formed at different densities and moisture contents (Chaney 2010).

In order to determine in the present experiment the effect of moisture content on the behavior of thermal properties of wood briquettes, they were humidified in a climatic test chamber (KPK 200/FEUTRON) at 20^{o}C and 90% RH. The moisture content of each briquette was determined according to the same method (SR EN 13183-1-2003/AC-2004), the density was obtained by using the stereometric method and the thermal conductivity and volumetric heat capacity were measured with KD2 Pro analyzer. The briquettes moisture content was increased from *EMC* up to the maximum moisture the briquettes could absorb and measurements of thermal properties could be performed.

The present paper deals only with the determination of briquettes specific heat, while the models applied to the determination of briquettes thermal conductivity were explained in detail in the paper reported by Sova et al.

According to Guo et al. (2012), a packed bed of pellets can be simplified by assuming it as continuous homogeneous porous system with effective thermal properties. Similarly, for a porous material that consists of solid particles and gas, as briquettes can be regarded, the effective volumetric heat capacity (ρc_p) is approximated by the following equation:

$$\rho c_p = (1-P_w)\rho_w c_{p\,w} + P_w \rho_{air} c_{p\,air} \quad (1)$$

where: c_p is the effective specific heat of briquettes ($Jkg^{-1}K^{-1}$), c_{pw} ($Jkg^{-1}K^{-1}$) and c_{pair} ($Jkg^{-1}K^{-1}$) are specific heats of wood particles and gas (air), ρ (kgm^{-3}), ρ_w (kgm^{-3}) and ρ_{air} (kgm^{-3}) are the densities of briquettes, wood particles and air, P_w is the volume fraction of gas in the wet porous material (wet porosity).

Guo et al. (2012) also considered that the effective specific heat of bulk pellets does not differ from the specific heat of a single pellet since the density of air is much smaller than the density of solid particles.

The effective specific heat of briquettes can be therefore expressed from Eq (1) as:

$$c_p = \frac{(1-P_w)\rho_w c_{p\,w} + P_w \rho_{air} c_{p\,air}}{\rho} \quad (2)$$

The wet porosity is described by Eq (3), (Hunt et al. 2008):

$$P_w = \frac{(1-V\%_{bw})P_d}{1-V\%_{bw}P_d} \quad (3)$$

where: $V\%_{bw}$ is the volume fraction of the bound water in the cell wall and P_d is the dry porosity.

The dry porosity is obtained from:

$$P_d = \frac{\rho_{cw_d} - \rho_d}{\rho_{cw_d} - \rho_{air}} \quad (4)$$

where: ρ_d (kgm^{-3}) is the oven dry density of briquettes, $\rho_{cw_d} = 1540 \mathrm{kgm}^{-3}$ is the density of the cell-wall substance (Siau 1995) and $\rho_{air} = 1.193 \mathrm{kgm}^{-3}$ is the density of air at 20°C (Incropera et al. 2007).

The volume fraction of the bound water, $V\%_{bw}$, is calculated as a function of the moisture content from the following equation (Hunt et al. 2008):

$$V\%_{bw} = \frac{MC\,\rho_{cw_d}}{\rho_{bw} + MC\,\rho_{cw_d}} \quad (5)$$

where: $\rho_{bw} = 1115 \mathrm{kgm}^{-3}$ is the density of the bound water (Hunt et al. 2008).

The density of wood particles, ρ_w, is determined using the rule of mixtures. Thus:

$$\rho_w = (1-V\%_{bw})\rho_{cw_d} + V\%_{bw}\rho_{bw} \quad (6)$$

According to Siau (1995) the specific heat of wood increases significantly with moisture content. When the moisture content is less than 5% the specific heat of wood may be calculated from the rule of mixtures (Siau 1995) as:

$$c_{pw} = \frac{1260 + 4185\,MC}{1+MC} \quad (7)$$

where: MC<0.5, t=30°C, the specific heat of oven dry wood at 30°C is $1260 Jkg^{-1}K^{-1}$ (Siau 1995), the specific heat of free water at 30°C is $4185 Jkg^{-1}K^{-1}$ (Siau 1995).

Specific heat also increases with temperature and according to Siau (1995) the specific heat of the dry cell wall may be calculated as:

$$c_{pw} = 1260\left[1 + 0.004\left(t^oC - 30\right)\right] \quad (8)$$

where: t°C is the temperature range from 0°C to 100°C.

The temperature at which specific heat of briquettes was measured was 20°C. Therefore, taking into account Eq (8) and the variation of the specific heat of water with temperature, Eq. (7) becomes:

$$c_{pw} = \frac{1209.6 + 4181MC}{1 + MC} \tag{9}$$

If the moisture content of wood increases above that mentioned in Eq (7), being in the range 5% and 24%, Siau (1995) suggests another relationship, based on a previous investigation, according to which an excess in specific heat must be taken in consideration. It corresponds to approximately 418Jkg^{-1}K^{-1}. Thus, Eq (7) changes into:

$$c_{pw} = \frac{1260 + 4185\,MC + 1674\left(MC - 0.05\right)}{1 + MC} \tag{10}$$

After rearranging terms, Eq (10) becomes:

$$c_{pw} = \frac{1176 + 5859\,MC}{1 + MC} \tag{11}$$

where: *MC* ranges from 0.05 to 0.3 and *t*=30^{o}C.

At *t*=20^{o}C, Eq (11) may be written as:

$$c_{pw} = \frac{1128 + 5808.5\,MC}{1 + MC} \tag{12}$$

The aforementioned equations for the determination of the specific heat are applied to the briquettes with *MC*=0% and *MC*=*EMC*. In this moisture content range, the voids between chips remain almost unchanged. When the moisture content increases above *EMC*, alongside with wood fiber swelling the magnitude and the number of the voids between chips increase too. A considerable increase in the briquette overall dimensions is noticed and for that reason the wet porosity increases, even if the moisture content increases and thus a decrease of porosity would be expected. Accordingly, in the range of moisture contents above *EMC* the wet porosity is not anymore calculated with Eq (3). In this range of moisture contents the wood cell has a larger lumen that includes the chips interspaces. The new length of the wood cell is determined from the proportionality between cell volume and briquette volume in oven dry conditions and the current moisture content conditions. It may be calculated from the following equation (Sova et al.):

$$L' = \left(\frac{V_b}{V_{b_d}}\right)^{1/2} \tag{13}$$

where: *L'* is the overall cell dimension, V_b is the briquette volume at current *MC*, V_{bd} is the briquette volume in oven dry conditions. The new lumen length, *a'*, is calculated from the equality (Sova et al.):

$$a' - a = L' - L \tag{14}$$

where: *a* is the lumen length of the wood cell, *L* is the cell dimension at a certain *MC*. The lumen length, *a*, is determined from the dry porosity (Siau 1995):

$$a = \left(P_d\right)^{1/2} \tag{15}$$

The dimension *L* is obtained from the dry and wet porosities as follows:

$$L = \left(\frac{P_d}{P_w}\right)^{1/2} \tag{16}$$

Detailed calculation of the overall cell dimension, *L*, is shown in (Sova et al.). Accordingly, the wet porosity of the briquettes with *MC>EMC* is determined from Eq (17):

$$P_w' = \frac{a'^2}{L'^2} \tag{17}$$

In Eq (2), the specific heat of air at *t*=20^{o}C is c_{pair} =1006.86Jkg^{-1}K^{-1} (Incropera et al. 2007).

RESULTS AND DISCUSSION

From the twenty briquettes subjected to thermal conductivity and specific heat measurements, four briquettes were removed from the range of data because they broke down during humidification. The most affected were those drilled both parallel and perpendicularly to their axis. Therefore, the measurements were further performed only on 16 briquettes.

The average moisture contents of the humidified briquettes were 5.95% (*EMC*), 12.2%, 13.1%, 16.3%, 17.6%, 21% and 22.7%, dry basis. Commercial wood pellets and briquettes have a

typical *EMC* of 4-8%, wet basis (Guo 2013).

The results were divided in two groups, one group comprising the briquettes drilled in parallel with the axis and the other group comprising the briquettes drilled perpendicularly to the axis.

Fig. 1 shows the experimental and modeled effective specific heat values corresponding to the longitudinally drilled briquettes with *MC*=0% and with *MC*=*EMC*, as function of the briquettes density. It was decided to plot the specific heat with respect to the briquettes density rather than with respect to their moisture content. The increase in moisture content from 0% to the *EMC* resulted in the linear increase of density and specific heat, which is in agreement with the statement of Siau (1995) regarding wood, that the specific heat increases significantly with moisture content. On the same figure, the specific heat of wood, described by Eqs (9) and (12), as function of density is represented too. The effective specific heat values are almost the same with the specific heat values of wood in this range of moisture contents. The low coefficients of determination (R^2) can be explained by the low number of data in this range of moisture contents. The scatter in the experimental results indicates possible differences in the local density of briquettes.

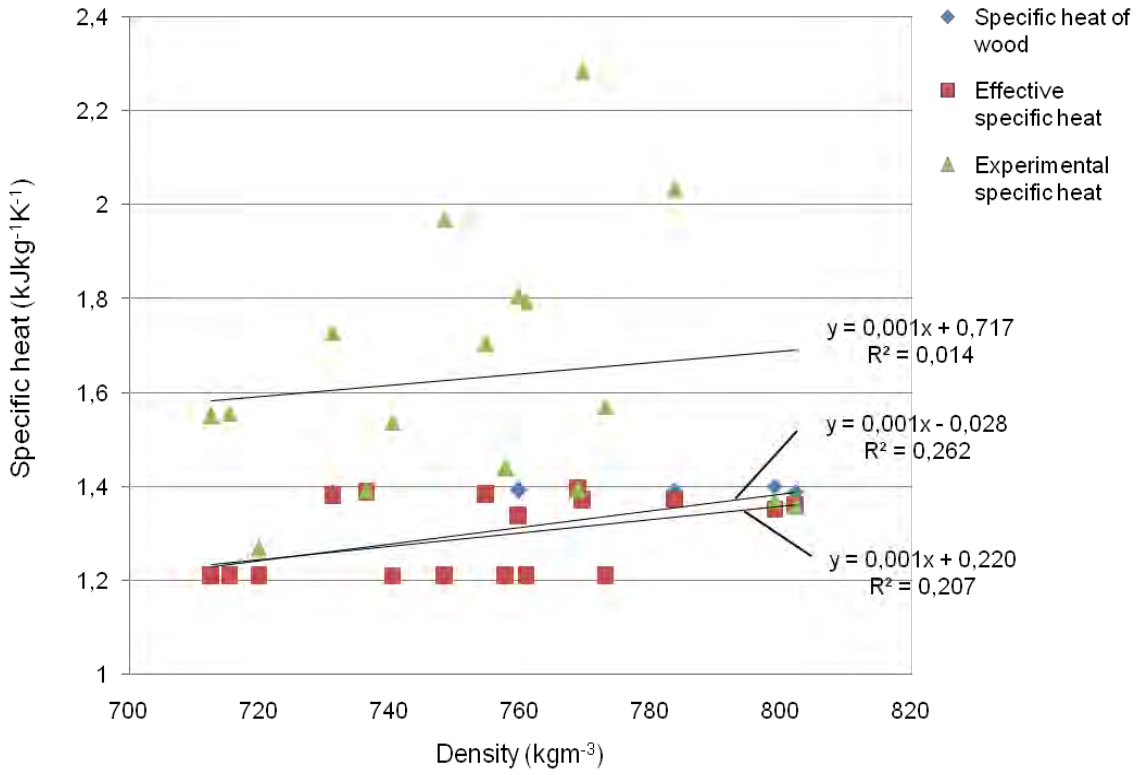

Fig. 1.
Experimental and calculated effective specific heat of longitudinally drilled briquettes as a function of density for MC≤EMC.

The experimental and modeled effective specific heat results corresponding to the radially drilled briquettes with *MC*=0% and with *MC*=*EMC*, as function of the briquettes density are indicated in Fig. 2. The figure also shows the results of the specific heat of wood calculated with Eqs (9) and (12). The results of the effective specific heat of briquettes and specific heat of wood are very similar to those represented in Fig. 1. They increase with density increase in a linear regression. The experimental results are again scattered and they increase very slightly with density increase.

Fig. 3 indicates the experimental and modeled effective specific heat values of the longitudinally drilled briquettes with *MC*>*EMC* as linear function of briquettes density. The increase in moisture content determined the decrease of the density because of the increase in the voids between the chips and the increase of the specific heat. The specific heat is not so much influenced by the increase in the voids as it is influenced by the increase of the moisture content. Only at moisture contents exceeding approximately 20%, the experimental results of the specific heat start to decrease because of the increase in the voids magnitude. The same ascendant trend has the specific heat of

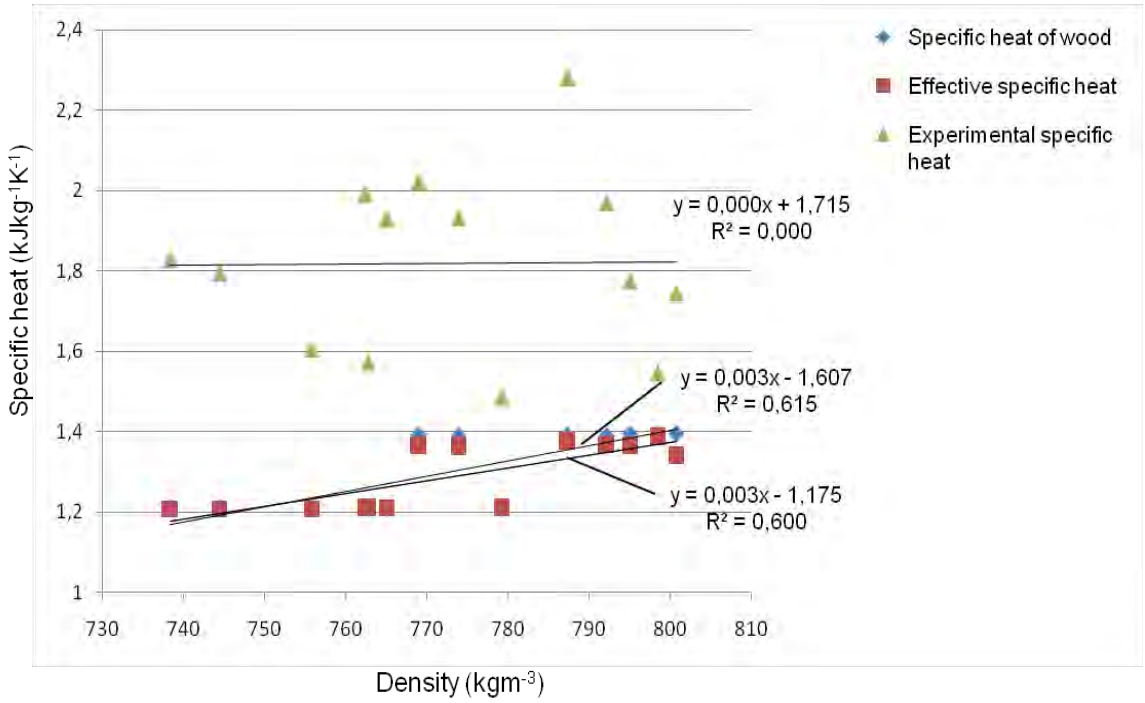

Fig. 2.
Experimental and calculated effective specific heat of radially drilled briquettes as a function of density for MC≤EMC.

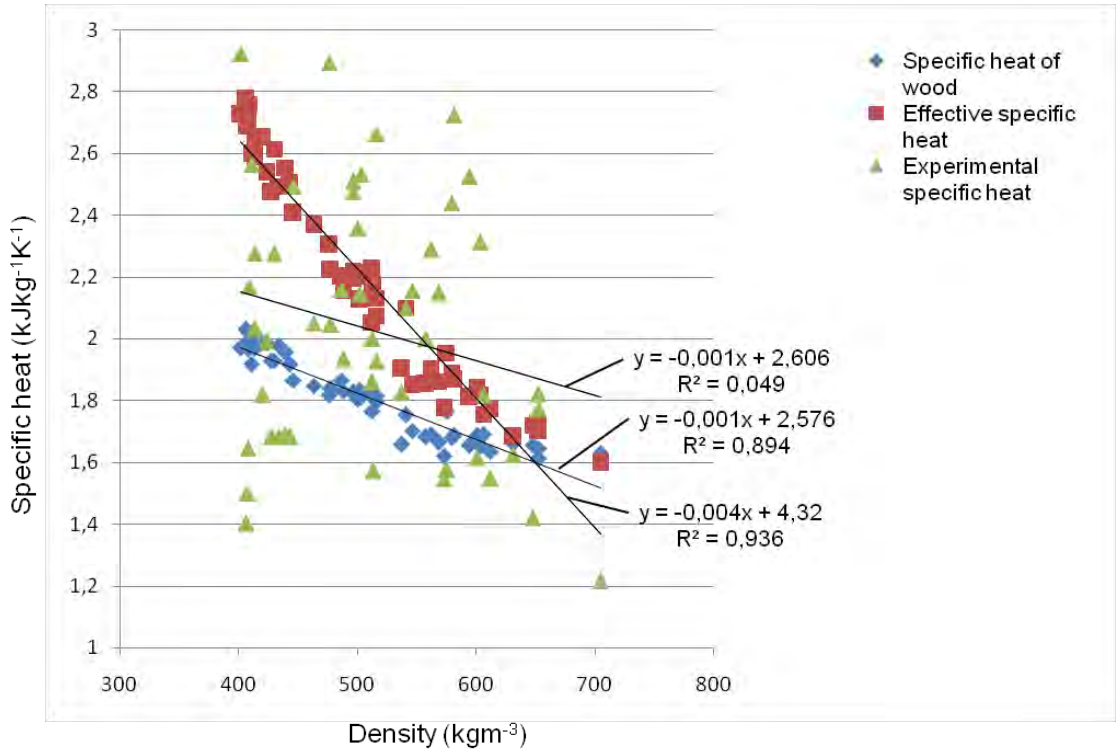

Fig. 3.
Experimental and calculated effective specific heat of longitudinally drilled briquettes as a function of density for MC>EMC.

wood. Comparing the specific heat results with the thermal conductivity results, the trend is different; i.e. the briquettes thermal conductivity decreased with moisture content increase, because the density decreased and the influence of the increasing voids became predominant (Sova et al.). It also can be physically explained by the fact that the decrease in the heat transfer by conduction determines the

increase in the energy stored in the briquette and less heat is required to raise the temperature by the same amount.

Fig. 4 indicates the experimental and modeled effective specific heat results corresponding to the radially drilled briquettes with *MC>EMC*, as function of the briquettes density.

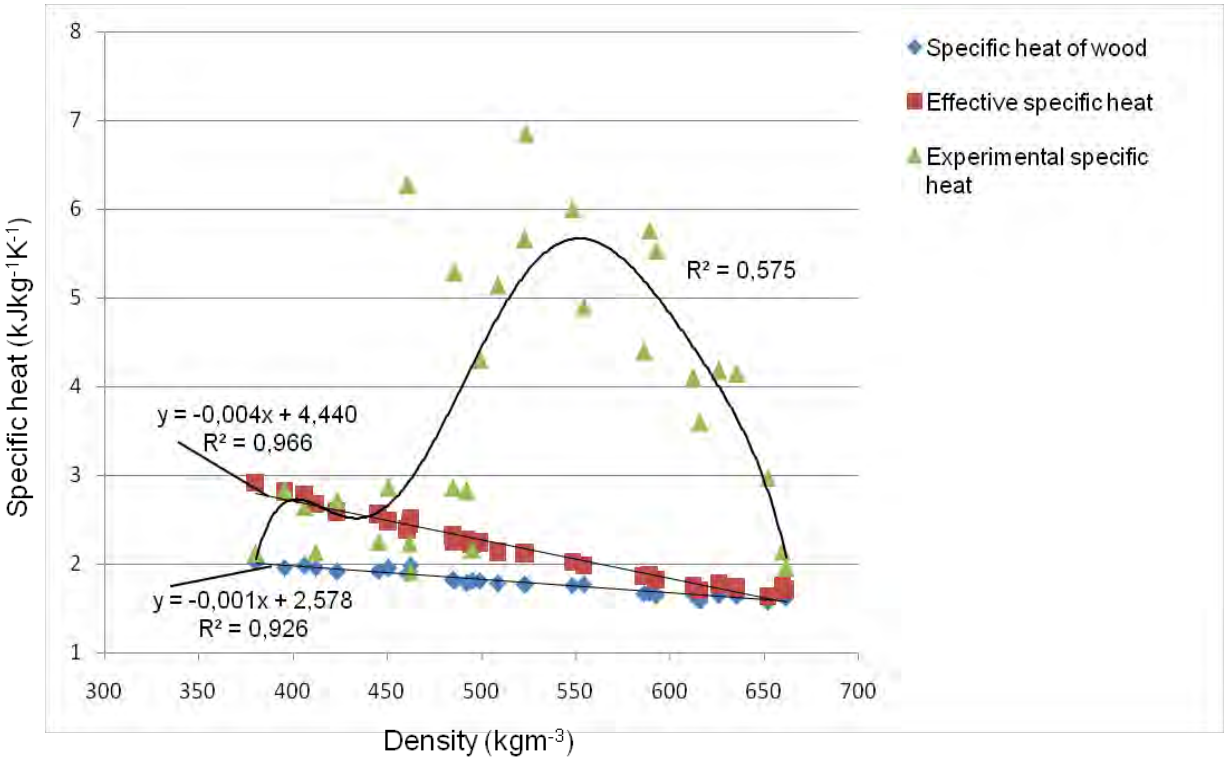

Fig. 4.
Experimental and calculated effective specific heat of radially drilled briquettes as a function of density for MC>EMC.

The increase in briquettes moisture content determined the decrease of their density and the linear increase in the effective specific heat. The specific heat of wood has the same trend and also is in linear regression with density. The experimental specific heat results increased with moisture content increase and with density decrease from 650kgm^{-3} to about 500kgm^{-3}; instead they decreased with density decrease starting with about 500kgm^{-3}. The variation of experimental specific heat with density is polynomial. Sova et al. indicated the same polynomial variation of the experimental thermal conductivity results with respect to the density of briquettes. They explained that the wood fiber swelling was more important on the briquettes length than in radial direction due to the wood swelling characteristics and that the effect of moisture content on specific heat, and thermal conductivity as well, was significant for the briquettes with the density ranging from 650kgm^{-3} to about 500kgm^{-3}. As the briquettes density decreased below 500kgm^{-3}, the influence of the voids became preponderant on both specific heat and thermal conductivity.

The experimental specific heat values are higher in case of the radially drilled briquettes than in case of the longitudinally drilled briquettes, thus depending on the measurement direction. The ratio of their average values is 1.7 (3.29kJkg^{-1}K^{-1} : 1.936kJkg^{-1}K^{-1}). On the other hand, the average values of the modeled specific heat of radially drilled briquettes and longitudinally drilled briquettes are in a ratio of 1 (1.976kJkg^{-1}K^{-1} : 1.973kJkg^{-1}K^{-1}). This shows that the modeled specific heat values are not sensitive to the measurement direction, perpendicular or parallel to the briquettes axis. It is to observe that the average experimental specific heat value of the longitudinally drilled briquettes is very close to the average modeled specific heat values. The experimental results of specific heat are therefore depending on moisture content, density and measurement direction.

The briquettes moisture content ranged from 0 to 24.5%, dry basis and the density ranged from 330kgm^{-3} (at maximum *MC*) to 802kgm^{-3} (at *EMC*). The typical range of moisture contents of wood used for fuel is from 5% to 20% (Ragland and Aerts 1991).

The specific heat of longitudinally drilled briquettes ranged from 1.217kJkg^{-1}K^{-1} to 3kJkg^{-1}K^{-1} and that of radially drilled briquettes from 1.485kJkg^{-1}K^{-1} to 6.843kJkg^{-1}K^{-1}. Guo et al. (2012)

determined effective specific heat values of wood pellets with the moisture content between 1.7 and 9% that ranged from 1.074 to 1.253kJkg^{-1}K^{-1}, increasing with moisture content linearly. The bulk densities ranged from 650 to 675kgm^{-3}. The specific heat of dry wood pellets was found by the same authors to be 1.01±0.05 (kJkg^{-1}K^{-1}). In the present paper the measured specific heat of dry briquettes ranged from 1.3 to 1.99kJkg^{-1}K^{-1} (the density ranged from 720 to 763kgm^{-3}). The specific heat of dry wood at 20^{o}C, as earlier mentioned in the paper, is 1.21kJkg^{-1}K^{-1}.

Pauner and Bygbjerg (2007) assumed in their paper a specific heat of 2.2kJkg^{-1}K^{-1} for studying self-heating of biofuel pellets.

Sjöström and Blomqvist (2014) measured the thermal properties of wood pellets at elevated temperatures using the transient plane source technique. For bulk densities ranging from 502 to 693kgm^{-3} and *MC*=6.6%, dry basis, the specific heat ranged from 1.35 to 1.63kJkg^{-1}K^{-1}. They also measured the specific heat of a single pellet with the density 1290kgm^{-3} at 2.9% and 11.7% moisture content and obtained the values 1.44kJkg^{-1}K^{-1} and 1.77kJkg^{-1}K^{-1}, respectively.

Fasina and Sokhansanj (1995) reported specific heat values of alfalfa pellets ranging from 1.636 to 2.021kJkg^{-1}K^{-1} within the moisture content range of 7.5 to 18%, wet basis and at 30^{o}C temperature. They used the line heat source method to obtain bulk thermal conductivity and thermal diffusivity. The specific heat of pellets was calculated from values of thermal conductivity, thermal diffusivity and bulk density.

Chaney (2010) measured the specific heat of newspaper briquettes by using the dual probe heat-pulse method over a range of densities (175-375kgm^{-3}) and obtained the mean value of 1.612kJkg^{-1}K^{-1} in a range of values between 1.2kJkg^{-1}K^{-1} and 2.3kJkg^{-1}K^{-1}. He assumed that the specific heat of briquettes was constant across the density range tested, because the contribution of the porosity had an insignificant effect on specific heat.

The experimental results reported by the above mentioned authors are comparable to those obtained for the longitudinally drilled briquettes, as described in this paper. As regards the results of the radially drilled briquettes, they are much higher than those reported in the above mentioned papers. The literature offers no mention that any other author would have measured the thermal properties of pellets or briquettes perpendicularly to their axis. Therefore, the research must be continued with more measurements at different temperatures and moisture contents in order to completely justify the results obtained in the experiment described within this paper.

CONCLUSIONS

According to experiments and models, the effective specific heat of briquettes increased with density increase, when the moisture content increased from 0% to the equilibrium moisture content. The same conclusion can be drawn for both measurements, in parallel and perpendicularly to the briquettes axis. When the moisture content increased from the equilibrium moisture content to the maximum moisture content the density decreased and the specific heat increased. According to the experiments made in parallel with the briquettes axis, the specific heat is not so much influenced by the increase in the voids as it is influenced by the increase of the moisture content. Only at moisture contents exceeding approximately 20%, the experimental results of the specific heat start to decrease because of the increase in the voids magnitude. For the experiments made perpendicularly to the briquette axis, the results showed the polynomial increase of the specific heat with moisture content increase and with density decrease from 650kgm^{-3} to about 500kgm^{-3}. Then, the specific heat decreased in a polynomial regression with moisture content increase and with density decrease from about 500kgm^{-3}. In this case the wood fiber swelling was more important on the briquettes length than in radial direction due to the wood swelling characteristics and the effect of moisture content on specific heat was significant for the briquettes with the density ranging from 650kgm^{-3} to about 500kgm^{-3}. As the briquettes density decreased below 500kgm^{-3}, the influence of the voids became preponderant on the specific heat. The experimental specific heat values are higher in case of the radially drilled briquettes than in case of the longitudinally drilled briquettes, thus depending on the measurement direction. The ratio of their average values is 1.7. The average experimental specific heat value of the briquettes measured in parallel with the axis is very close to the average modeled specific heat value.

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SECTION 3.
MECHANICAL WOOD PROCESSING AND SURFACE QUALITY

RESOURCE UTILISATION IN A PRODUCTION CELL FOR LAMINATED VENEER PRODUCTS

Lars BLOMQVIST
Linnaeus University, Department of Building Technology
SE-351 95 Växjö, Sweden
Tel: +46-(0)470-70 89 34, E-mail: lars.blomqvist@lnu.se

Jimmy JOHANSSON
Linnaeus University, Department of Forestry and Wood Technology
SE-351 95 Växjö, Sweden
E-mail: jimmy.johansson@lnu.se

Steffen LANDSCHEIDT
Linnaeus University, Department of Forestry and Wood Technology
SE-351 95 Växjö, Sweden
E-mail: steffen.landscheidt@lnu.se

Henrietta NILSON
Linnaeus University, Department of Entrepreneurship and organization
SE-351 95 Växjö, Sweden
E-mail: henrietta.nilson@lnu.se

Abstract

The concept of productivity is often used to determine how well resources are used in an operation, and it is usually determined as the ratio of what is consumed in the production. Laminated veneer products are considered complicated products often taking complex shapes, using a raw material with high variation, and requiring machining processes that create scrap material that needs to be handled. Therefore, maintaining high productivity in industries producing such products may become challenging. This study reports on productivity measurements in a production cell consisting of an adhesive, pressing and a processing station. The study seeks to increase the understanding of production-related problems in this industry. This research has been based on productivity measurement as well as on interactive discussions between researchers and workers. Measurement of cycle times indicated bottlenecks in the processing cell. The discussion led via cycle times, processing residues and chatter marks to an examination of the foundation and rigidity of the CNC-machine in the processing cell. The study indicated that the performance of the CNC machine did not correspond to expectations. The machine was too weak to handle the required output in an efficient manner. Thus, there is a need to determine the performance expected before a machine or machine group is purchased. An update of the existing purchasing literature and its dissemination will support the crystallization of the purchasing process as a way forward to support the industry.

Key words: *CNC; robot; stiffness; vibration.*

INTRODUCTION

The concept of productivity is often used to determine how well resources are being used in an operation. It is usually determined as the ratio of what is produced to what is consumed in the production (Olhager 2000). Productivity can be given as Overall Equipment Effectiveness (OEE), which is calculated by multiplying the availability, performance, and quality (Bicheno et al. 2011). Laminated veneer products (LVPs) are considered complicated products often taking complex shapes, using a raw material with high variation (Blomqvist 2015) and requiring machining processes that create scrap material that then needs to be handled. Therefore, maintaining high productivity in industries producing such products may become challenging. Flexible automation in the manufacturing of LVPs has been used as a working hypothesis to examine the possibility of developing the wood furniture industry. A production cell that produces LVPs was chosen to carry out these studies, seeking to increase understanding of production-related problems in this industry. The aim of this particular study was to create an understanding of production-obstructive events in this industry, by investigation the potential for improvement in the selected production cell with the help of

cycle time (CT) registration and root cause analysis (RCA). RCA is a method of asking the question "why?" until the root cause is discovered (Petersson et al. 2009).

METHODS

The study has involved a combination of productivity measurement and interactive discussions between researchers and workers regarding its methods (Archibald 2016).

Case description

The production cell contained two robot-automated cells with an intermediate press station that was handled manually. These persons were also engaged in the support and control of the robot-automated cells. In the first robot cell, veneers were taken from different magazines by the robot, adhesive was spread on the veneers via a bead/string application and the veneers were packed in bundles in preparation for moulding. This sub-cell is here called the adhesive station. In the press cell, the staff took the bundles of veneers with adhesive and placed them in form-pressing tools where the laminates were moulded. There were three presses, each with an identical pressing tool. This sub-cell is here called the press station. After the press station, the staff moved the moulded seat shells to a conveyor belt that served as a cooling station and as interim storage prior to the second automated robot cell, where a robot took the seat shell from the conveyor belt and positioned it in the CNC machine for processing. After the CNC-processing, the robot took the shells to different sanding units and finally placed them on a conveyor belt. This sub-cell is here called the processing station. After the processing station, the shell was taken manually and placed in a box for lacquering, as shown in Fig. 1. There were other activities concerning the seat shell before and after its processing in the production cell, but these activities were not investigated in this study.

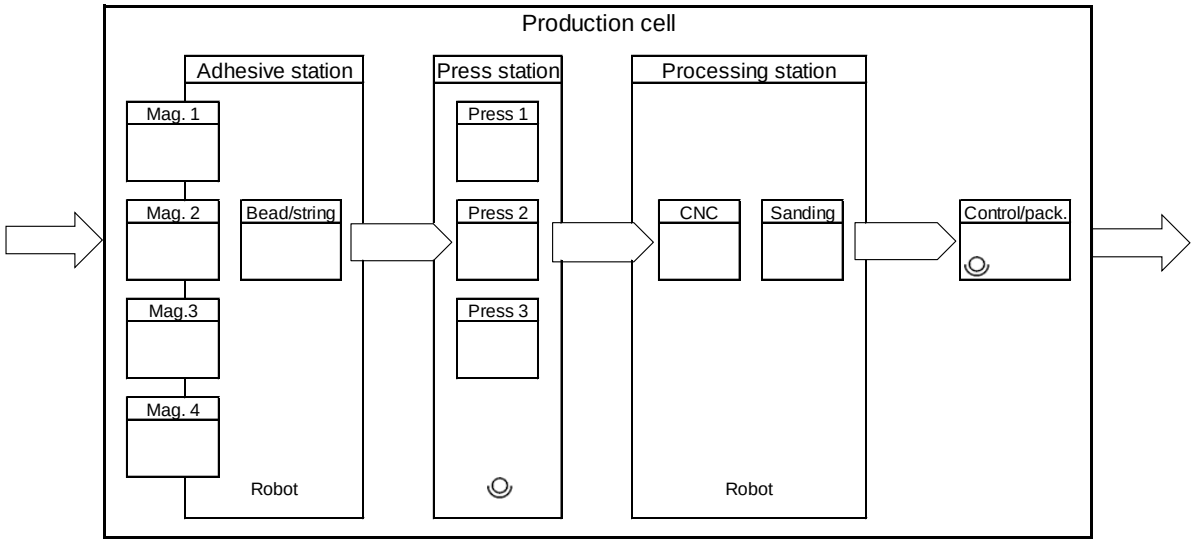

Fig. 1.
The different stages in the production cell where the seat shells were moulded and processed.

Data collection

The resource utilisation was controlled in various parts of the production cell by recording the cycling times (CTs), which were used to calculate the maximum possible production and effects of various improvements. The researcher observed and asked questions on several occasions to gain an understanding of the production cell. A workshop was arranged in which all employees at the production cell participated, together with management and researchers. The discussion processed CT causes with RCA. This resulted in further measurements and analysis of the machine setup regarding stiffness of the floor and machine head.

EMPERICAL FINDINGS AND ANALYSIS

The CNC machine was recognized as the production cell's limiting factor, i.e. the bottleneck, in the production of the seat shells. The CT for a seat shell was 172 seconds in the CNC-machine compared with 128 seconds in the adhesive station, 137 seconds in the press station (calculated as: [349+62]/3=137), and 158 seconds for robot handling in the processing station (20+138=158) (Table 1).

Table 1

Cycling times of different production steps

Place	CT (sec)
Adhesive station	128
Press station	
• Correcting a bundle of veneers	27
• Loading of press and pressing time	349
• Movement from press to conveyer belt	62
Processing station	
• Robot gripping and positioning	20
• CNC-milling	172
• Sanding	138

CNC-milling

The CNC-milling was divided into two steps: a rough machining to remove unnecessary material and rough mill the contour with a feeding speed of 9,000–12,000mm per minute, and a fine milling of the contour with a fine milling cutter, with a feeding speed of 2,000–5,000mm per minute, depending on the severity of the processing.

The rough contour milling gave a lot of residual material. Optimization of the first step of the CNC-milling in conjunction with a new rough milling cutter with greater length and diameter could shorten the time and reduce the amount of residual material. Calculations indicated that this could shorten the CT by 15 seconds, so that the CT of the CNC-milling could be shortened from 172 seconds to 157 seconds (Table 2).

The feed rate of the fine milling cutter was low, due to chatter marks after the contour milling (Fig. 2). The chatter marks became larger with faster feeding and complicated the sanding of the milled contour.

Fig. 2.
Chatter marks on the contour milled edge after fine milling.

An increase in the feed rate of the fine milling would lead to a further shortening of the CT of up to approximately 12 seconds in the CNC-milling. Together with the previously mentioned improvement this could mean a reduction in CT to 145 seconds. The bottleneck would then be shifted to the other two cycles of the process station which together took 158 seconds. The time for sanding could, however, be reduced if the seat shell edge had a better surface after the CNC-milling. It was, therefore, reasonable to expect a CT of 158 seconds as the limiting factor if the CNC-program were optimized regarding rough milling and a further reduction down to 145 seconds if the chatter marks could be radically reduced (Table 2).

Table 2

Cycling times in different productions steps with capacity calculations related to various alternatives for improvement

Place	Alt.	CT (sec)
Adhesive station		128
Press station		137
Processing station – robot		158
Processing station – CNC	1	172
Processing station – CNC	2	157
Processing station – CNC	3	145

Vibration in relation to chatter marks

It appeared that the CNC machine vibrated and that such vibrations increased in amplitude with increasing feed rate, which could explain the chatter marks and with the effect of feed rate on the

chatter marks. It also emerged that the vibrations had been reported and were to be addressed during service of the CNC-machine when a linear bearing had to be replaced.

A poor foundation was suggested to be the probable cause of the vibration. However, the manufacturer of the examined CNC-machine had not specified any special requirements of the floor on which the machine would be set up. Nevertheless, a manufacturer of a similar CNC-machine (6 ton) advocates floors to withstand a "*minimum unit load capacity of 20 000N/m^2 over the machining centre-bearing surface"* to withstand load and stress (Venus 2007). Examination of the floor resulted in an estimate that the concrete thickness was 100mm with unknown material underneath.

Investigation of floor and machine stiffness

The floor's dynamic stiffness and the natural frequency of the mounting of the tool in the machine were measured, using a hammer equipped with a built-in force sensor that excited the test points. An accelerometer was used to measure the response in the same direction as the excitation with the hammer, i.e. perpendicular to the floor (Fig. 3). Measurements of the natural frequency of the machine head were made in its X, Y and Z directions (Fig. 4).

Fig. 3.
Measuring point at the upper rear machine foot of the CNC machine.

Fig. 4.
Measurements of the machine head in the CNC machine. Excitation took place with the hammer on the opposite side of the spindle in which accelerometers were placed in the X and Y plane (horizontal). The vertical excitation took place together with the accelerometer in the Z-direction (vertical).

The floor's mobility was at its highest, -110 dB (ref. 1m/Ns) in the low frequency ranges (around 10 Hz) where the machine was located. The machine head stiffness was different in different directions, being weakest in the X-direction. The spindle mobility was at its lowest, -90 dB. There was thus a difference of at least 20 dB between the floor and the machine head. Converted to static stiffness, this means that the floor was about 10 times stiffer than the machine head, and that the greatest reason for the machine head's pliancy at low frequencies was the machine's own weakness, and, to a smaller extent, the weakness of the floor (Olsson 2016).

DISCUSSION AND CONCLUSIONS

The need to fix the bundles of veneer after the adhesive station can be considered to be an unnecessary step because it adds no value to the flow of the production of the seat shell. That step ought to be eliminated by ensuring correct bundles. Two suggestions emerged during the study: to include a vibrating tray that shakes the pack into the correct position, or to ensure the correct position by refining the handling of the veneers from the different veneer magazines together with a vision system that ensured the final positioning. The productivity in the production cell would not be affected by this extra step. The same applies to the other two steps in the press station, although, these two steps add value to the process. There is generally a need to study the value of the flow in and out of the production cell to create a better basis for decisions.

Optimisation of the milling in the CNC-machine would mean less processing residue, less vibration and shorter CTs. If the vibrations were minimised to give fewer and smaller chatter marks, then the sanding would also be easier and the total capacity would increase.

Knowledge of the machine performance is obviously important when purchasing, but it is also significant during usage. Although the condition of the floor was found to have less impact than the machine's own pliancy, it is important to consider a possible relocation of the machine to ensure the optimal performance of the foundation.

One way to reduce vibrations would be to optimize the CNC-program. To reduce the forces during acceleration and deceleration through smoother transitions would help to avoid sudden jerks and stops. This is applicable especially in the machine head's X-direction in which the machine head was weakest. This would help to reduce the out-swings occurring after, for example, an abrupt stop.

To handle a machine with lower performance than desired it is important to understand its limitations. Requirements of, for example, surface finish could control various levels of processing, but if the limits are exceeded, the service needs are affected.

When purchasing, it is important to have an understanding of the forces to which the machine will be exposed. Therefore, it should be natural that prospective buyers require an inspection protocol and definition of the machine's optimal performance conditions to ensure purchase of the right machine. It is important to have a clear criterion in the procurement of processes that affect surface finish. For example, an understanding of how the natural frequencies (maximum and minimum resonance frequency in the different directions in relation to the position of the machining tool affect its own frequency) and the specific type of processing makes it possible to determine the maximum feed rate for a particular surface finish, which could ultimately be a type of productivity measurement for the machine.

Sjöberg and Höglund (1997) developed a guideline with a focus on the wood product industries for the purchase of complex machinery. Their handbook covers, among others topics, pilot studies, requirement specifications, quotations, delivery and investment cost calculations. However, there are reasons to update and process the handbook to reflect the needs of new business models and technologies. The findings in the present study give an indication of the parameters which should be included in an updated handbook.

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MECHANIC-MATHEMATICAL MODEL FOR INVESTIGATIONS OF THE FORCED SPATIAL VIBRATIONS OF WOOD SHAPER AND ITS SPINDLE, CAUSED BY UNBALANCE OF THE CUTTING TOOL

Georgi VUKOV
University of Forestry – Sofia, Faculty of Forest Industry
Blvd. Kliment Ohridski 10, 1797 Sofia, Bulgaria
Tel: 359 2 91907/285, E-mail: givukov@ltu.bg

Zhivko GOCHEV
University of Forestry – Sofia, Faculty of Forest Industry
Blvd. Kliment Ohridski 10, 1797 Sofia, Bulgaria
E-mail: zhivkog@ltu.bg

Valentin SLAVOV
University of Chemical Technology and Metallurgy – Sofia, Faculty of Metallurgy
Blvd. Kliment Ohridski 8, 1756 Sofia, Bulgaria
E-mail: valslav@abv.bg

Pavlin VICHEV
University of Forestry – Sofia, Faculty of Forest Industry
Blvd. Kliment Ohridski 10, 1797 Sofia, Bulgaria
E-mail: p_vitchev@abv.bg

Valentin ATANASOV
University of Forestry – Sofia, Faculty of Forest Industry
Blvd. Kliment Ohridski 10, 1797 Sofia, Bulgaria
E-mail: vatanasov_2000@yahoo.com

Abstract

A mechanic - mathematical model of wood shaper and its spindle, developed by the authors, is presented in this work. The model provides the opportunity to explore the forced space vibrations of this type of machinery, caused by unbalance of the cutting tool. It takes into account the characteristics in the construction of wood shapers. In this model the wood shaper and its spindle are regarded as rigid bodies, which are connected by elastic and damping elements with each other and with the motionless floor. The model takes into account the necessary mass, inertia, elastic and damping properties of the elements of the considered system. It includes all needed geometric parameters of this system. A necessary system of matrix differential equations is compiled and analytical solutions are presented. Numerical solutions can be obtained with their help by using the parameters of a specific machine.

Key words: *wood shapers; forced vibrations.*

INTRODUCTION

The high productivity of the operation of wood shapers, and the precision and quality of their production are the main requirements to them. The main factors determining the implementation of these requirements are the status and serviceability of the cutting tool of these machines (Filipov 1977, Grigorov 1985). Some fixed regulations to their geometry, materials for production, ways of assembly of the instruments to the shaft and etc, are required. The practice in the exploitation of wood shapers indicates that one of the common problems in their use is the presence of unbalance (disbalance) of their cutting tools. The causes for rising of the unbalance are: wrong or incorrect installation of the instrument on the shaft; uneven wear or damage of the tool; accumulation of superposition in separate parts of the instrument; occurrence of gaps and the etc. The influence of the unbalance of the cutting tool on the machine has to be studied in order to be done some research. The machine can be seen as a mechanical vibrating system with known characteristics in this research (Amirouche 2006, Angelov and Slavov 2010, Coutinho 2010).

It is known that there may be few cases of unbalance. The cutting tool is statically unbalanced if its center of gravity does not lie on the axis of rotation, but it is the main inertial axis. The static

unbalance can be detected with static tests. The cutting tool is dynamically unbalanced if its center of gravity lies on the axis of rotation, but it is not the main inertial axis. The dynamic unbalance cannot be detected with static tests. The cutting tool can be statically and dynamically unbalanced at the same time, of course. This paper examines the static imbalance of the cutting tool.

The presence of unbalance of the cutting tool generates variable loads during the operation of the wood shapers. These loads are transmitted to the spindle and by its two bearing units reach to the machine's body. On the other hand, vibrations, generated by other elements of the machine, reach the spindle and the cutter back through the bearing units. It is clear that the characteristics of the bearing units (stiffness, damping properties, etc.) are important for the interaction between the spindle with the cutter and the machine's body, and consequently, for the work of the whole machine.

To sum up, it is clear that the wood shaper and its spindle are appropriate to be regarded as rigid bodies. They are connected by elastic and damping elements with each other and with the motionless floor. These elastic and damping elements are four vibration isolators between the machine and the floor, as well as both bearing units of the spindle. This approach has been used in previous works of the authors. The free space vibrations are examined (Vukov et al. 2016) and the natural frequencies and mode shapes of the free spatial vibrations of real wood shaper are investigated numerically (Vukov et al. 2016). The free damped spatial vibrations are discussed (Vukov et al. 2016) and some numerical studies are conducted (Vukov et al. 2016).

OBJECTIVE

The main objective of this study is to develop a mechanic - mathematical model of a wood shaper and its spindle, which gives the opportunity for exploration the forced space vibrations of this type of machinery, caused by unbalance of the cutting tool. The model refers to wood shapers with lower placement of the spindle. The model renders in account the construction's characteristics of this class of wood shapers. The developed model allows making numerical investigations by using parameters of real machines.

MATERIAL, METHOD, EQUIPMENT

The wood shapers with lower placement of the spindle that is commonly used in the practice of the forestry industry (Filipov 1977, Obreshkov 1996) is examined in the proposed study. Analysis of their construction shows the strong influence of the unbalance of the cutting tool on the functioning of the whole machine. Fig. 1 shows the general view, and Fig. 2 – the spindle with its bearing units and fitted cutter.

Fig. 1.
Wood shaper – general view.

Fig. 2.
Spindle with bearing units and with cutter.

The wood shaper and its spindle are regarded as rigid bodies in the following discussions. They are connected by elastic and damping elements with each other and with the motionless floor. These elastic and damping elements are four vibration isolators between the machine and the floor, as well as the two bearing units of the spindle. The static unbalance of the cutting tool, which is frequent in practice, is considered in this study. This unbalance is modeled with the introduction of a centrifugal force acting on the cutter. The value of centrifugal force is determined by the magnitude of the unbalanced mass, the eccentricity (the distance from the axis of rotation to the center of gravity of the tool) and the square of the angular velocity.

A mechanic - mathematical model of wood shapers with lower spindle is built for studying its forced spatial vibrations, caused by unbalance of the cutting tool. The model is shown in Fig. 3.

The following symbols are used:
m_1, m_2 – mass of the wood shaper and its spindle;
$\boldsymbol{I_1}$, $\boldsymbol{I_2}$ – inertia moment tensors of the wood shaper and its spindle;
$\boldsymbol{c_{x1i}}$, $\boldsymbol{c_{y1i}}$, $\boldsymbol{c_{z1i}}$, $i = 1, 2, 3, 4$ – elastic coefficients of the vibroisolators between the machine and the floor;
$\boldsymbol{b_{x1i}}$, $\boldsymbol{b_{y1i}}$, $\boldsymbol{b_{z1i}}$, $i = 1, 2, 3, 4$– damping coefficients of the vibroisolators between the machine and the floor;
$\boldsymbol{c_{x2i}}$, $\boldsymbol{c_{y2i}}$, $\boldsymbol{c_{z2i}}$, $i = 5, 6$ – elastic coefficients between the machine and the spindle;
$\boldsymbol{b_{x2i}}$, $\boldsymbol{b_{y2i}}$, $\boldsymbol{b_{z2i}}$, $i = 1, 2, 3, 4$ – damping coefficients between the machine and the spindle .

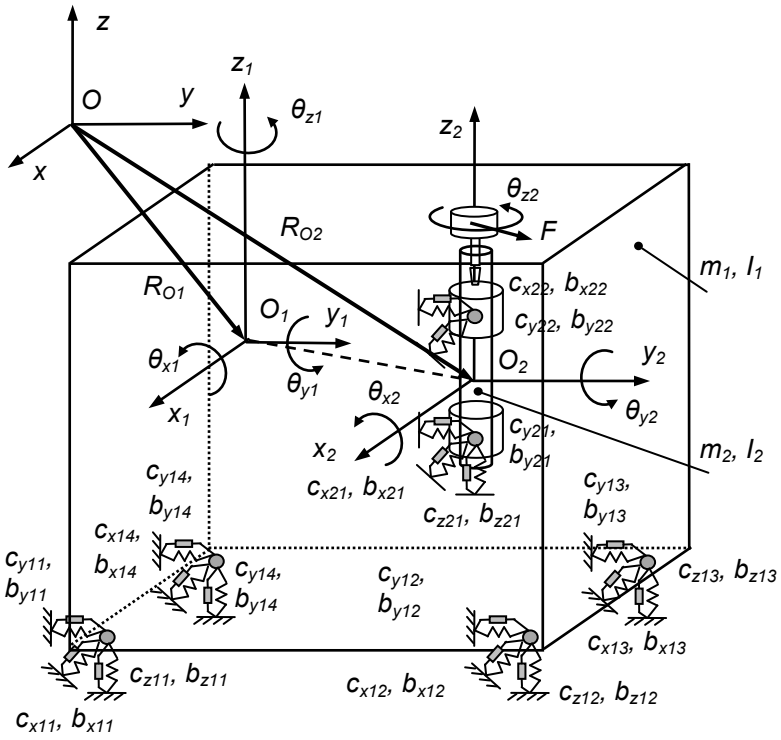

Fig. 3.
Mechanic-mathematical model of the wood shaper and its spindle.

The vector of the generalized coordinates is (Fig. 3)

$$q = \left[x_1\ y_1\ z_1\ \theta_{x1}\ \theta_{y1}\ \theta_{z1}\ x_2\ y_2\ z_2\ \theta_{x_2}\ \theta_{y_2}\ \theta_{z_2}\right]^T . \tag{1}$$

The matrixes of the transition in small vibrations between the local coordinate systems of the bodies and the reference coordinate system have the form

$$A_i^0 = \begin{bmatrix} 1 & -\theta_{zi} & \theta_{yi} & x_i \\ \theta_{zi} & 1 & -\theta_{xi} & y_i \\ -\theta_{yi} & \theta_{xi} & 1 & z_i \\ 0 & 0 & 0 & 1 \end{bmatrix} \qquad i = 1,2 . \tag{2}$$

The vector of the position of the center of mass of the relevant body is determined with

$$R_{Ci}^0 = A_i^0 . r_{Ci} = \begin{bmatrix} l_{Cx} + x_i + l_{Cz} . \theta_{yi} - l_{Cy} . \theta_{zi} \\ l_{Cy} + y_i - l_{Cz} . \theta_{xi} + l_{Cx} . \theta_{zi} \\ l_{Cz} + z_i + l_{Cy} . \theta_{xi} - l_{Cx} . \theta_{yi} \\ 1 \end{bmatrix} \qquad i = 1,2 . \tag{3}$$

where: $r_{Ci} = \left[l_{Cx} \quad l_{Cy} \quad l_{Cz} \right]^T$ is the vector of the position of the center of mass in the local coordinate system.

The vector of absolute linear velocity of the center of mass of the respective body is calculated as follows

$$V_{Ci}^0 = \frac{dR_{Ci}^0}{dt} = \begin{bmatrix} \dot{x}_i + l_{Cz} . \dot{\theta}_{yi} - l_{Cy} . \dot{\theta}_{zi} \\ \dot{y}_i - l_{Cz} . \dot{\theta}_{xi} + l_{Cx} . \dot{\theta}_{zi} \\ \dot{z}_i + l_{Cy} . \dot{\theta}_{xi} - l_{Cx} . \dot{\theta}_{yi} \\ 0 \end{bmatrix} \qquad i = 1,2 . \tag{4}$$

The vector of absolute angular velocity of the respective body, projected in the local coordinate system, has the form

$$\Omega_i^i = \begin{bmatrix} \dot{\theta}_{xi} \\ \dot{\theta}_{yi} \\ \dot{\theta}_{zi} \\ 0 \end{bmatrix} \qquad i = 1,2 . \tag{5}$$

The deduction of the kinetic energy and potential energy of the system is convenient to be made with a symbolic method and modern software (Mathematica, MATLAB).

The kinetic energy of the mechanical system is determined with

$$E_K = \sum_{i=1}^{2} \left(\frac{1}{2} . \left[\dot{R}_{Ci}{}^T \quad \dot{\Theta}_i{}^T \right]^T . \begin{bmatrix} m_{RRi} & \\ & I_{\Theta\Theta i} \end{bmatrix} . \begin{bmatrix} \dot{R}_{Ci} \\ \dot{\Theta}_i \end{bmatrix} \right), \tag{6}$$

where: $m_{RRi} = \begin{bmatrix} m_i & 0 & 0 \\ 0 & m_i & 0 \\ 0 & 0 & m_i \end{bmatrix}$; $\mathbf{I}_{\Theta\Theta i} = \begin{bmatrix} I_{xxi} & -I_{xyi} & -I_{xzi} \\ -I_{xyi} & I_{yyi} & -I_{yzi} \\ -I_{xzi} & -I_{yzi} & I_{zzi} \end{bmatrix}$;

$\dot{R}_{Ci} = \left[\dot{x}_{Ci} \quad \dot{y}_{Ci} \quad \dot{z}_{Ci} \right]^T$; $\dot{\Theta}_i = \left[\dot{\theta}_{xi} \quad \dot{\theta}_{yi} \quad \dot{\theta}_{zi} \right]^T$.

Potential energy is defined by

$$E_P = E_{P1} + E_{P2} = \left(\sum_{k=1}^{4} \frac{1}{2} c_k . \left(\delta r_k^{01} \right)^2 + \sum_{k=1}^{2} \frac{1}{2} c_k . \left(\delta r_k^{12} \right)^2 \right) + \sum_{i=1}^{2} E_{PGi} , \tag{7}$$

where: $\delta r_k^{01} = R_1 + U_1^0 . r_k^{01} - r_k^{01}$,

$\delta r_k^{12} = \left(R_1 + U_1^0 . r_k^{12} - r_k^{12} \right) - \left(R_2 + U_2^0 . r_k^{21} - r_k^{21} \right)$,

$R_i = \left[x_i \quad y_i \quad z_i \right]^T \quad i = 1,2$ - vector of the position of the beginning of the mobile (related with the body) coordinate system relative to the fixed coordinate system,

δr_k^{01} – the deformation of the elastic elements between the base (marked conditionally with "0") and the body 1,

δr_k^{12} – the elastic deformation of the elements between the two bodies.

The differential equations which describe the free vibrations are deduced by using the Lagrange's method. This method provides the best opportunities.

$$\frac{d}{dt}\left(\frac{\partial E_K}{\partial \dot{q}}\right)-\left(\frac{\partial E_K}{\partial q}\right)+\frac{\partial F_b}{\partial \dot{q}}+\frac{\partial E_P}{\partial q}=Q \quad , \tag{8}$$

where: E_K and E_P are respectively the kinetic and the potential energy of the systems. F_b is the energy dissipation or dissipative function. Q is the vector of generalized forces.

The obtained system of differential equations, which describes the forced spatial vibrations of the mechanical system, is

$$M_{12\times12}.\ddot{q}_{12\times1}+B_{12\times12}.\dot{q}_{12\times1}+C_{12\times12}.q_{12\times1}=Q_{12\times1}. \tag{9}$$

The matrix in these equations which characterizes the mass-inertial properties of the mechanical system is M, and the elastic properties – C. $B(\dot{q})$ is the matrix that characterizes the damping properties of this system and Q presents generalized forces

$$M=\left[a_{ij}\right], \qquad a_{ij}=\frac{\partial^2 E_K}{\partial \dot{q}_i.\partial \dot{q}_j}, \tag{10}$$

$$C=\left[c_{ij}\right], \qquad c_{ij}=\frac{\partial^2 E_P}{\partial q_i.\partial q_j}. \tag{11}$$

The matrix $B=\left[b_{m,n}\right]$ is obtained by substituting the elements of the matrix C – $c_{m,n}$, with $b_{m,n}$.

$$B=\left[b_{ij}\right], \qquad b_{ij}=\frac{\partial^2 F_b}{\partial \dot{q}_i.\partial \dot{q}_j}. \tag{12}$$

The vector of generalized external forces has the form

$$Q=\begin{bmatrix}0 & 0 & 0 & 0 & 0 & 0 & Q_{F(3\times1)}{}^T & Q_{Q(3\times1)}{}^T\end{bmatrix}^T. \tag{13}$$

where:

$$Q_F=\begin{bmatrix}F.\cos(\omega.t)\\ F.\sin(\omega.t)\\ 0\end{bmatrix}, \tag{14}$$

$$Q_Q(F)=U_i^{\Omega 0T}.\left(\tilde{r}_{Pi}^{0T}.Q_F\right), \tag{15}$$

$$U_i^{\Omega 0\,T}=\begin{bmatrix}1 & 0 & \theta_{y1}\\ 0 & 1 & -\theta_{x1}\\ 0 & \theta_{x1} & 1\end{bmatrix}^T, \tag{16}$$

$$\tilde{r}_{Pi}^{0T}=\begin{bmatrix}0 & I_{Piz}^0 & -I_{Piy}^0\\ -I_{Piz}^0 & 0 & I_{Pix}^0\\ I_{Piy}^0 & -I_{Pix}^0 & 0\end{bmatrix}, \tag{17}$$

The receiving of general solutions of the system (9) is connected with the determination of the initial conditions of motion $q\ (0)$ and $\dot{q}(0)$. These initial conditions depend on the type of motion of the system.

The general solutions of the system of differential equations, written in a matrix form in harmonious kind of disturbing forces and initial conditions $t=0,\ q(0)=q_0,\ \dot{q}(0)=\dot{q}_0$, are

$$q(t) = \sum_{r=1}^{12} \frac{2}{g_r^2 + h_r^2} [G_r M \dot{q}(0) + (-\alpha_r G_r M + \beta_r H_r M \quad + G_r B) q(0)] \; .e^{-\alpha_r t} .\cos \beta_r t +$$
$$+ \sum_{r=1}^{12} \frac{2}{g_r^2 + h_r^2} [H_r .M.\dot{q}(0) + (-\alpha_r .H_r .M - \beta_r .G_r .M + H_r .B).q(0)] \; .e^{-\alpha_r t} .\sin \beta_r t + \quad (18)$$
$$+ Re\{ \sum_{k=0}^{n} \sum_{r=1}^{12} \frac{2}{g_r^2 + h_r^2} \frac{\alpha_r .G_r + \beta_r .H_r + i.k.\Omega .G_r}{\omega_r^2 - k^2 .\Omega^2 + i.2.k.\sigma_r .\omega_r .\Omega} Q.e^{ik\Omega t} \}$$

where:

$$g_r = -2\alpha_r \left(V_r^T .M.V_r - W_r^T .M.W_r \right) - 4\beta_r V_r^T .M.W_r + V_r^T .B.V_r - W_r^T .B.W_r ;$$
$$h_r = 2\beta_r \left(V_r^T .M.V_r - W_r^T .M.W_r \right) - 4\alpha_r V_r^T .M.W_r + 2V_r^T .B.W_r ; \quad (19)$$
$$G_r = g_r L_r + h_r R_r ; \quad L_r = V_r .V_r^T - W_r .W_r^T ;$$
$$H_r = h_r L_r - g_r R_r ; \quad R_r = V_r .W_r^T + W_r .V_r^T .$$

CONCLUSIONS

This study presents a mechanic - mathematical model of a wood shaper and its spindle, developed by the authors. The model is designed for investigation of the forced spatial vibrations of this type of machinery, caused by unbalance of the cutting tool. It takes into account the characteristics in the construction of wood shapers. In this model the wood shaper and its spindle are regarded as rigid bodies, which are connected by elastic and damping elements with each other and with the motionless floor. The model takes into account the necessary mass, inertia, elastic and damping properties of the elements of the considered system. It includes all needed geometric parameters of this system. A necessary system of matrix differential equations is compiled and analytical solutions are presented. Numerical investigations are carried out with them by using the parameters of a real machine. These investigations are presented in the next part of this study.

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NUMERICAL INVESTIGATIONS OF THE FORCED SPATIAL VIBRATIONS OF A WOOD SHAPER AND ITS SPINDLE, CAUSED BY UNBALANCE OF THE CUTTING TOOL

Georgi VUKOV
University of Forestry – Sofia, Faculty of Forest Industry
Blvd. Kliment Ohridski 10, 1797 Sofia, Bulgaria
Tel: 359 2 91907/285, E-mail: givukov@ltu.bg

Zhivko GOCHEV
University of Forestry – Sofia, Faculty of Forest Industry
Blvd. Kliment Ohridski 10, 1797 Sofia, Bulgaria
E-mail: zhivkog@ltu.bg

Valentin SLAVOV
University of Chemical Technology and Metallurgy – Sofia, Faculty of Metallurgy
Blvd. Kliment Ohridski 8, 1756 Sofia, Bulgaria
E-mail: valslav@abv.bg

Pavlin VICHEV
University of Forestry – Sofia, Faculty of Forest Industry
Blvd. Kliment Ohridski 10, 1797 Sofia, Bulgaria
E-mail: p_vitchev@abv.bg

Valentin ATANASOV
University of Forestry – Sofia, Faculty of Forest Industry
Blvd. Kliment Ohridski 10, 1797 Sofia, Bulgaria
E-mail: vatanasov_2000@yahoo.com

Abstract

This study presents the results of the numerical investigations of the forced spatial vibrations of a wood shaper and its spindle, caused by unbalance of the cutting tool. The paper is based on a specific mechanical - mathematical model, developed by the authors, which allows studying of vibrations of this type of machinery. In this model a wood shaper and its spindle are regarded as rigid bodies, which are connected by elastic and damping elements with each other and with the motionless floor. This study renders an account the mass, inertia, elastic and damping properties and geometric parameters of the machine. The results of the numerical investigations are presented. They are obtained through modern software and by using parameters of a particular machine.

Key words**:** *wood shapers; forced vibrations*.

INTRODUCTION

Unbalance check of the spindle and cutting tool of the woodworking shaper machine is a compulsory stage of the preparation of the machine for its putting in action. Monitoring and timely detection of occurred unbalance during machine operation, largely determines the effectiveness of its work (Filipov 1977, Obreshkov 1996). The study of unbalanced forces on the spindle and cutting tool of the woodworking shaper machine is essential for its proper putting in action and as well as to guarantee its normal operation. The unbalanced inertia forces of the instrument cause significant additional dynamic loads on the spindle bearings, and consequently, on the whole construction (Grigorov 1985). The result of this additional load decreases significantly the operational resource of the machine. Sometimes this may even cause emergency stop of the machine for imperative repairs. This leads to serious losses from downtime due to unplanned required repairs. Another possible result of the rising of the significant unbalanced inertia forces of the cutting tool of the machine is a violation of the accuracy and quality of its products.

The analysis of the unbalance influence of the cutting tool on the machine operation requires carrying out specific studies. The machine can be seen as a mechanical vibrating system with known characteristics in these researches (Amirouche 2006, Coutinho 2010). Furthermore, it is necessary to apply modern methods for studying of dynamic systems. The majority of modern methods for the

study of dynamic systems are oriented towards the use of computing technique, which allows obtaining the final results of the study in numerical form (Angelov and Slavov 2010). The solving a practical problem cannot be achieved directly by applying one or several numerical methods.It is usually too long and complicated to deal with the problem as it starts with putting the problem for solving and finishes with obtaining the final results of the calculations, which are applicable in practical implementation.Series of numerical investigations, measurements and experiments must be conducted, systematized and worked up. These investigations are based on specifically developed model of the studied machine.

Some different mathematical models of the studied machinery can be built for the same solved problems. They have to reflect as accurately as possible the character and manner of operation of the researched object. The choice of model depends on the specific tasks of the study. Appropriate numerical method can be applied just after analyzing all data output and choosing a suitable mathematical model. Each numerical method that associates the mathematical model with a numerical algorithm guarantees a certain accuracy of the results. These results are the primary database for solving the practical problems. All this refers to the research of work of the wood shaper machine and it is reflected in previous works of authors (Vukov et. al. 2016).

OBJECTIVE

The objective of this study is to conduct numerical investigations of the forced spatial vibrations of a wood shaper machine and its spindle, caused by unbalance of the cutting tool. The model refers to wood shapers with lower placement of the spindle. The obtained results are illustrated graphically in order to be analyzed more easily. The investigations are done on the basis of the developed by the authors concrete mechanic - mathematical model for the study of forced spatial vibrations of these types of wood shapers. The model is presented in the previous part of this work. The model renders in account the construction's characteristics of this class of wood shapers. The wood shaper and its spindle are regarded as rigid bodies, which are connected by elastic and damping elements with each other and with the motionless floor. These elastic and damping elements are four vibration isolators between the machine and the floor, and the two bearing units of the spindle. Static imbalance of the cutting tool is modelled. The calculations use the parameters of a real wood shaper.

MATERIAL, METHOD, EQUIPMENT

The first part of this study presents a built-up by the authors dynamic model of the machine. It gives a system matrix differential equations and appropriate analytical solutions. The numerical solutions which are obtained on their base are submitted in this part of the work. Parameters of the machine FD-3, produced in ZDM – Plovdiv, Bulgaria and put into practice, are used. These parameters are presented below.

The two bodies and the whole machine are modelled by software Solid Works in this part of the study. These models are shown respectively in Fig. 1, Fig. 2 and Fig. 3. The mass centre of the body 1 coincides with the centre of the local coordinate system of the body 1 and the centre of the reference coordinate system. The mass centre of the body 2 coincides with the centre of the local coordinate system of the body 2.

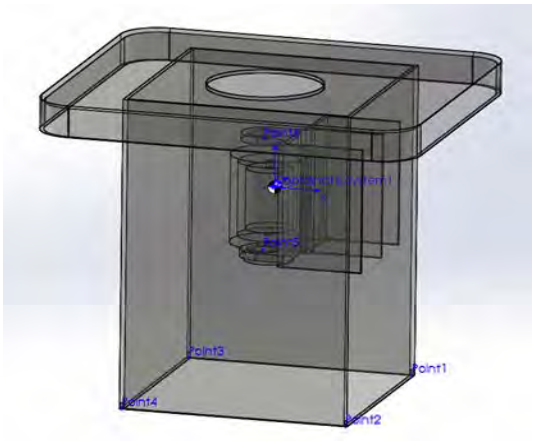

Fig. 1.
Body 1

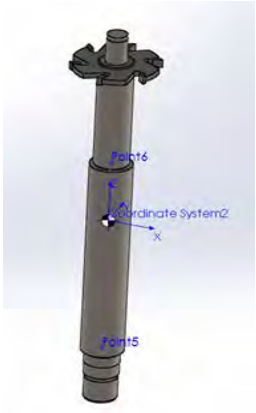

Fig. 2.
Body 2

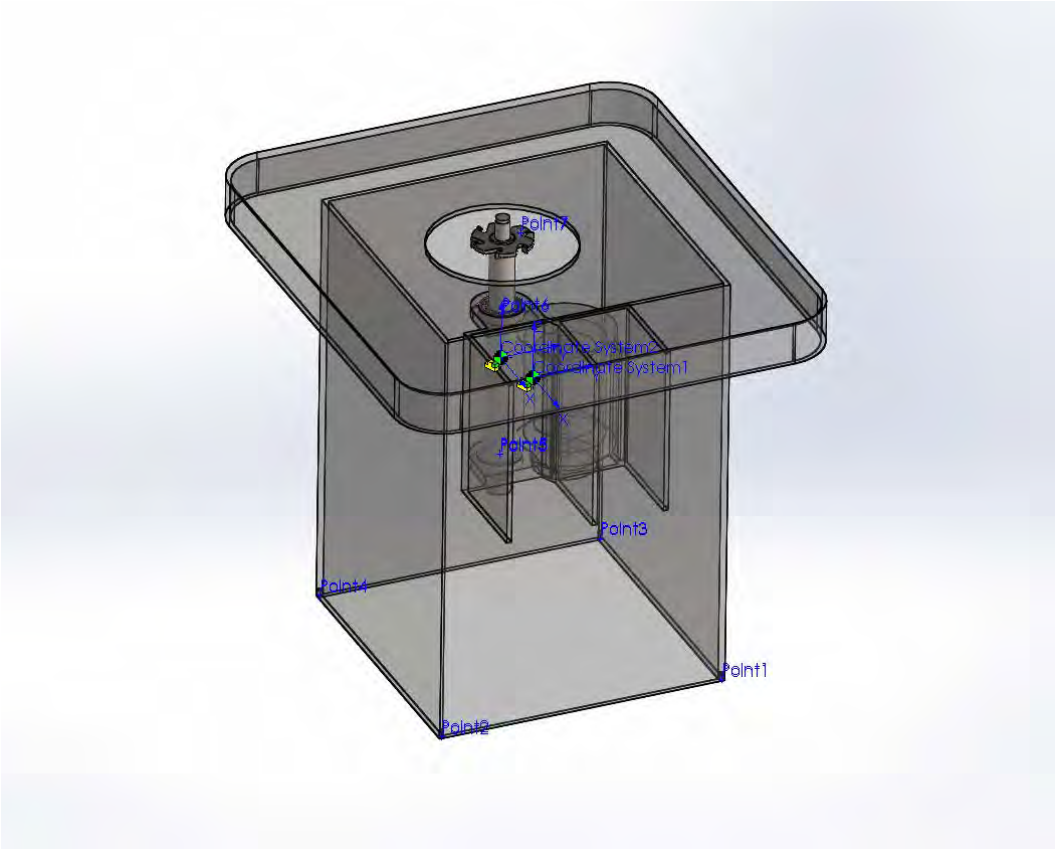

Fig. 3
Wood shaper.

The presented data below are used for the calculations. They are of the real machine FD-3 which is often used in practice.
Mass of ***the*** body 1 ***– m_1 = 303,43 kg***; ***the*** body mass 2 ***– m_2 = 11,12 kg***. Tensor of ***mass*** inertia ***moments*** of the body 1 relative to the local coordinate system of the body 1***, kg.m^2***

M1=303.43; M2=11.12;

$$I_1 = \begin{bmatrix} 37,2215 & -0,1064 & -0,0566 \\ -0,1064 & 38,6641 & -0,1915 \\ -0,0566 & -0,1915 & 34,8599 \end{bmatrix}$$

Tensor of ***mass*** inertia ***moments*** of the body 2 relative to the local coordinate system of the body 2***, kg.m^2***

$$I_2 = \begin{bmatrix} 0,2937 & 0 & 0 \\ 0 & 0,2937 & 0 \\ 0 & 0 & 0,0052 \end{bmatrix}$$

J1xx=37.2215; J1yy=38.6641; J1zz=34.8599;
J1xy=0.1064; J1xz=0.0566; J1yz=0.1915;
J2xx=0.2937; J2yy=0.2937; J2zz=0.0052; J2xy=J2xz=J2yz=0;

Coordinates of the pivot points of the body 1 to the centre of the coordinate system of the body 1***, m:***

p.1	p.2	p.3	p.4
$x = 0,287$	$x = 0,287$	$x = -0,303$	$x = -0,303$
$y = 0,279$	$y = -0,311$	$y = 0,279$	$y = -0,311$
$z = -0,579$	$z = -0,579$	$z = -0,579$	$z = -0,579$

Coordinates of the pivot points of the body 2 to the centre of the coordinate system of the body 1***, m:***

p.5	p.6
$x = -0,008$	$x = -0,008$
$y = -0,066$	$y = -0,066$
$z = -0,160$	$z = 0,151$

Coordinates of the pivot points of the body 2 to the centre of the coordinate system of the body 2***, m:***

p.5	p.6
$x = 0$	$x = 0$
$y = 0$	$y = 0$
$z = -0,214$	$z = 0,096$

Elasticity coefficients***, N/m***

cx011=cx012=cx013=cx014=350000;
cy011=cy012=cy013=cy014=350000;
cz011=cz012=cz013=cz014=800000;
cx125=cx126=1500000;
cy125=cy126=2250000;
cz125=4500000; cz126=0;

Damping coefficients, ***(N.s)/m***

bx011=bx012=bx013=bx014=980;
by011=by012=by013=by014=670;
bz011=bz012=bz013=bz014=470;
bx125=bx126=980;
by125=by126=670;
bz125=470; bz126=0;

Coordinates of the application point of the force of the unbalance (p. 7) in the coordinate system of the body 2

$$r_F = \begin{bmatrix} l_{Fx} \\ l_{Fy} \\ l_{Fz} \end{bmatrix} = \begin{bmatrix} -0,018 \\ 0,046 \\ 0,258 \end{bmatrix}$$

RESULTS AND DISCUSSION

Calculations have been made for three different values of the angular velocity of the spindle – 66 s^{-1}, 100 s^{-1} and 133 s^{-1} and at three values of the unbalance – 0.010 kg.m, 0.015 kg.m and 0.020 kg.m. Just a few of the results are represented here due to the limited place. The results illustrating vibrations of the machine body (coordinate q_1), as well as those of the cutter (coordinate q_7), are presented below. Fig. 4 shows the result for these coordinates when the unbalance is 0.010 kg.m for the three angular velocities of the spindle (66 s^{-1}, 100 s^{-1} and 133 s^{-1}). Fig. 5 shows the results at unbalance 0.015 kg.m for the same three angular speeds of the spindle, and fig. 6 - at unbalance of 0.020 kg.m.

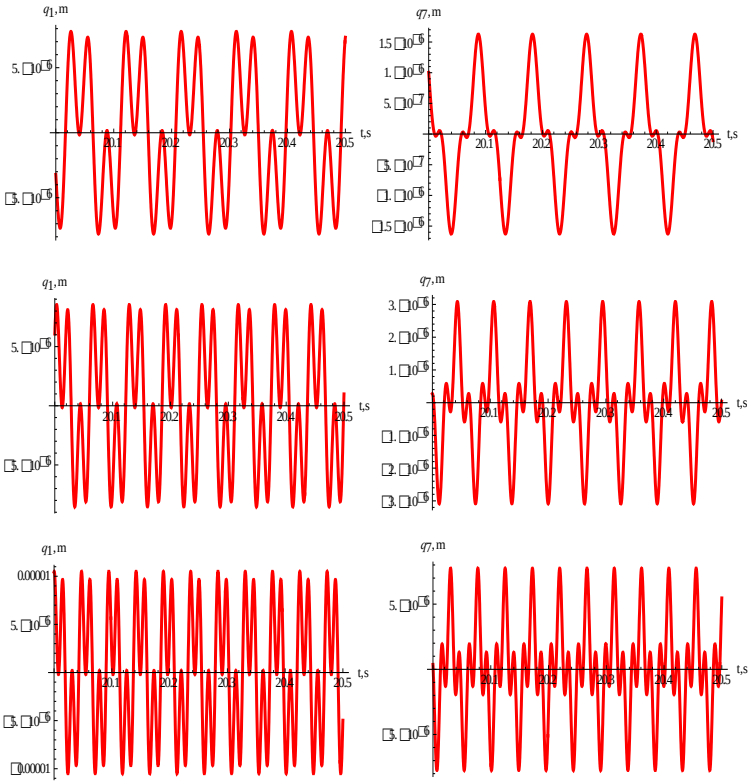

Fig. 4.
Results unbalance 0.010 kg.m

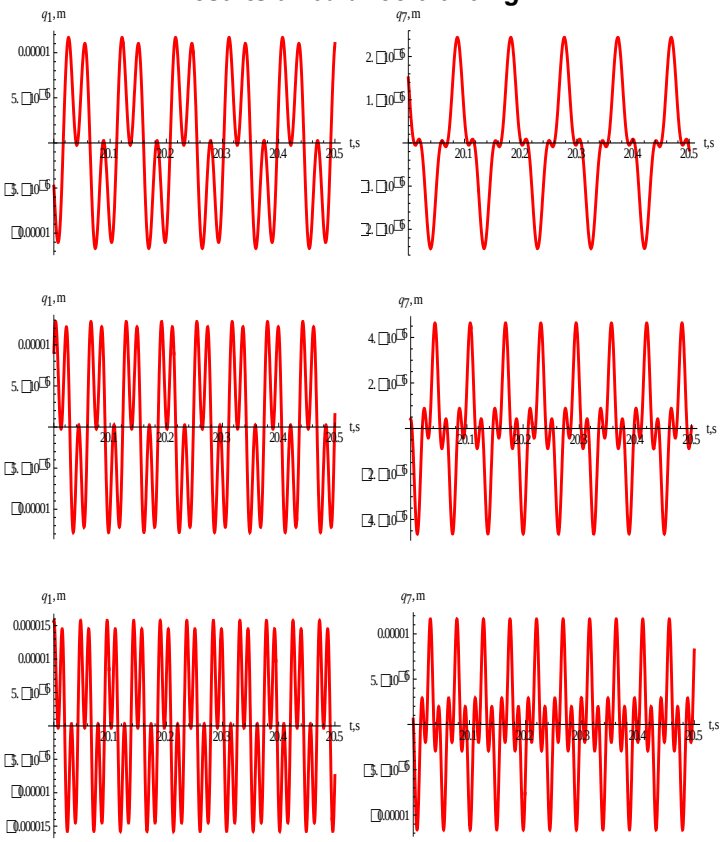

Fig. 5.
Results unbalance 0.015 kg.m

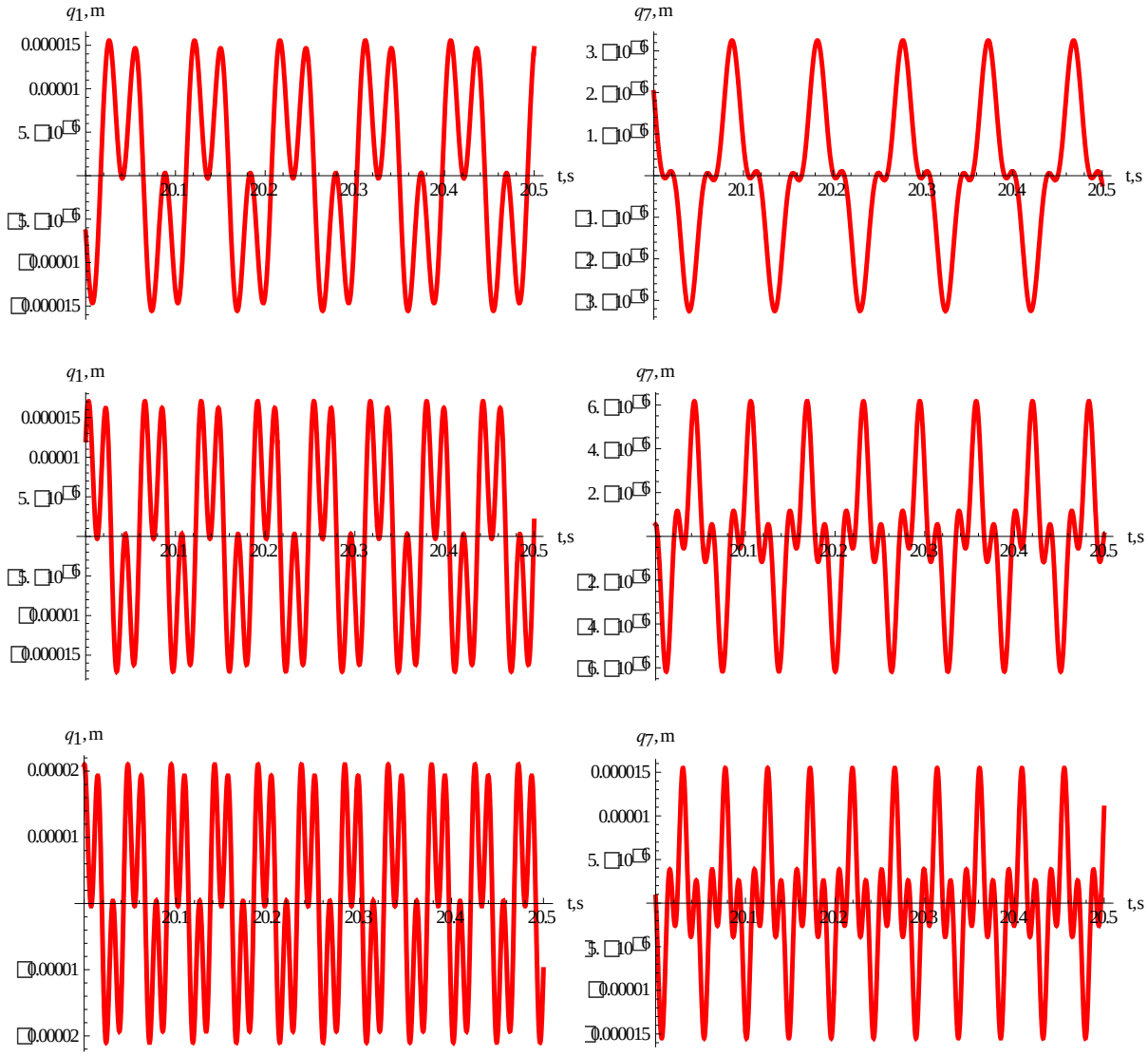

Fig. 6.
Results unbalance 0.020 kg.m

The analysis of the obtained results shows that presence of unbalance of the cutting tool of wood shaper generates intense spatial vibrations not only on the spindle, but on the whole machine. The amplitude of these vibrations increases greatly along with the increase of the value of unbalance and angular speed of the spindle. The specific values depend on the machine parameters and conditions of its work. It confirms the need for precise pre-balancing of the cutting tool, which is especially important in fast moving wood shapers. The results verify the applicability of the model developed by the authors for studying the vibration behavior of wood shapers.

CONCLUSIONS

The presented study investigates and illustrates the forced spatial vibrations of a wood shaper and its spindle, caused by unbalance of the cutting tool. The study is made numerically by using a modern software product. The calculations are performed on the base of a specific mechanical - mathematical model, which allows studying of vibrations of this type of machinery. The advantages of the model are in the consideration of the characteristics in the construction of these kinds of wood shapers. The model also includes parameters of elastic and damping elements of the structure. Numerical investigations are carried out by using the parameters of a real machine. The results of the conducted investigations allow analyzing the influence of static unbalance of the cutting tool on the vibration behavior of this machine. Raising of the reliability of the machine as well as the accuracy and quality of processing products is the final goal, which is meant.

ACKNOWLEDGEMENT

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TRACEABILITY AND ADAPTIVE PRODUCTION IN THE DIGITAL SAWMILL

Johan SKOG
RISE Reseach Institutes of Sweden, Division of Bioeconomy
Laboratorgränd 2, 931 77 Skellefteå, Sweden
Tel: 0046 70 617 6247, E-mail: johan.skog@ri.se

Mikael JACOBSSON
RemaSawco
Snickaregatan 40, 582 26 Linköping, Sweden
E-mail: mikael.jacobsson@remasawco.se

Anders LYCKEN
RISE Reseach Institutes of Sweden, Division of Bioeconomy
Drottning Kristinas Väg 67, 114 28 Stockholm, Sweden
E-mail: anders.lycken@ri.se

Abstract

The paper describes the development and the evaluation under industrial conditions of a fingerprint traceability system for Scots pine (Pinus sylvestris L.) and Norway spruce (Picea abies L.). It was found that 99% correct matches could be obtained while tracing 89% of all pine boards and 87% of all spruce boards.

This method can be used to connected production data along the whole production chain and will provide new information on the relationship between raw material properties and final product properties. The paper also elaborates on how such information will form the basis of the adaptive production control in the digital sawmills of the future.

Key words: *boards; digitalization; fingerprint; logs; sawmill; traceability.*

INTRODUCTION

Over the past decades, the sawmill industry has become more and more digitized. Nowadays, a wide range of sophisticated measurement systems are used for scanning logs and boards in different stages of the production chain. For example, many sawmills use X-ray-based log scanners to predict the grade of sawn goods before actual sawing takes place, thereby optimizing production by using the right logs for the right products. Sawn goods are scanned with optical systems or X-rays in order to determine dimensions and grade and to make decisions for the further processing.

The data collected by such instruments is however still used only to a limited extent. The scanning systems are primarily being used for making production decisions in the current production stage, for example an edging, cross-cutting, grading or sorting decision. Some information is also being logged into databases which enables follow-up of the production on batch level.

Data about individual logs or boards are however rarely being saved and reused later on in the process. The main reason for this is the difficulty of keeping track of the identity of logs and boards throughout the production chain. Literature suggest many different methods for keeping track of individuals between different stages of the production using additional ID marking equipment such as RFID transponders, ablation lasers or color markers (***eg.*** Uusijärvi 2010).

Another approach involves using the biological diversity of the wooden raw material to calculate unique fingerprints of each individual. These fingerprints can be identified in a later stage and information associated with the individual can be loaded from a database and reused. Tracing from logs to sawn boards has been described in literature (Flodin ***et al.*** 2008) and was made an industrial implementation by Skog ***et al.*** (2015).

The successful industrial implementation of a system capable of accurately and noninvasively tracing almost every log to boards has opened the doors to the digital sawmill industry of the future. In a currently ongoing research project, large amounts of connected sawmill production data will be gathered, forming big data describing the process. Such data give completely new opportunities to analyze and optimize the sawmill production.

OBJECTIVE

The main objective of the research present in this paper was to evaluate the traceability technology in an industrial environment and to elaborate on possible future uses of big data for adaptive production control in the digital sawmill.

MATERIAL, METHOD, EQUIPMENT

X-ray Log Scanning

Most industrial X-ray log scanners use a limited number (typically 1–4) of fixed measurement directions (*e.g.* Aune 1995; Pietikäinen 1996; Grundberg & Grönlund 1997). The most common solution on the market is two-directional X-ray log scanners producing two perpendicular radiographs as the log is fed through the scanner (Fig. 1). The X-ray radiographs of the log contain data describing the internal X-ray attenuation distribution of the log. From these data, it is possible to distinguish many different internal features of the log. Detection of knot structure has been presented by *e.g.* Pietikäinen (1996) and Grundberg & Grönlund (1998).

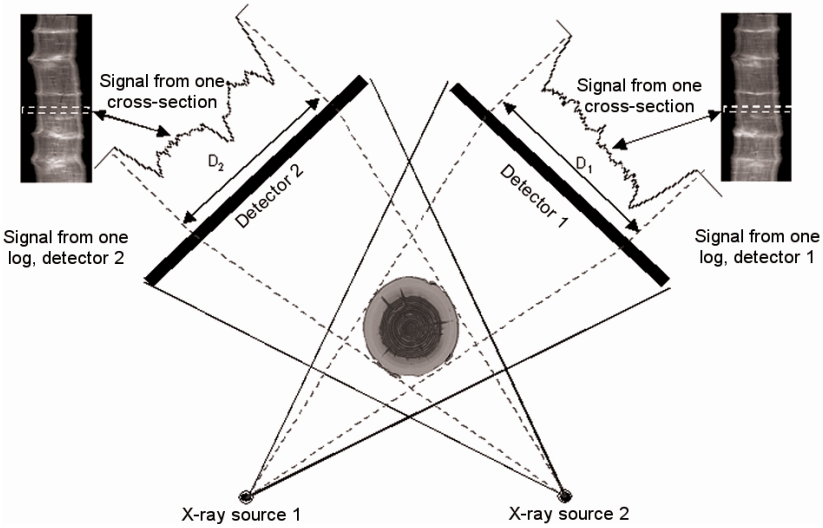

Fig. 1.
Schematic of the X-ray log scanner described by Grundberg and Grönlund (1997).

Board Scanning

Board scanners are vision systems that collect images of the sawn wood. By analysing these images, the board scanner extracts information about board features such as dimension, wane and knots. The RemaSawco RS-BoardScannerQ (Fig. 2) also includes a laser-based tracheid system that measures the grain angles across the board and uses this information to improve the knot detection.

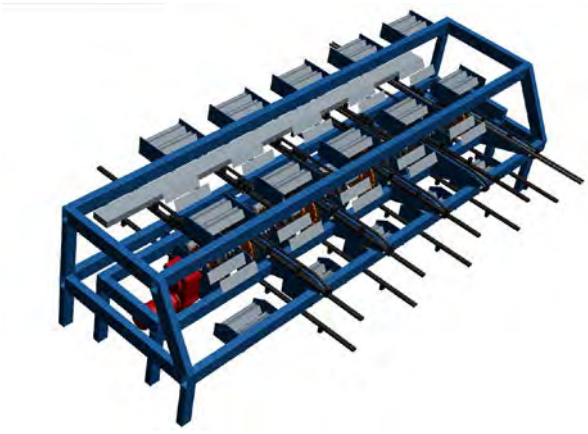

Fig. 2.
Board scanner with traditional vision system and laser-based tracheid measurements (Illustration by RemaSawco).

Data Collection

A total of 22 926 Norway spruce and Scots pine sawlogs harvested in mid Sweden were scanned using an X-ray log scanner of the brand RemaSawco RS-Xray (Grundberg & Grönlund 1997; RemaSawco 2017b). Out of these logs, 884 logs (338 Norway spruce and 546 Scots pine) of varying diameter had been manually numbered on both log ends and followed through the X-ray log scanner.

These 884 logs where then sawn into boards using a 2-ex sawing pattern, while keeping track of the log numbers. This resulted in a total of 1768 boards (676 Norway spruce and 1092 Scots pine), each board being individually numbered, thus estabilshing a true relationship between the logs identities and the corresponding board identities. After drying, all 1768 board were transported to another sawmill that had a board scanner of the brand RemaSawco RS-BoardScannerQ (RemaSawco 2017a). There, all boards were scanned while keeping track of the board numbers.

Altogether, this resulted in one data set containing individually matched X-ray log data and board scanner data of 338 Norway spruce and 546 Scots pine sawlogs and one reference data set contiaining X-ray data of the remaining 22042 logs. The purpose of the reference data set was to ensure that the developed algorithms would not create false positives.

Calculation of fingerprints

Fingerprint algorithms were developed based on the principles described by Flodin et al. (2008). A knot fingerprint was calculated for each log based on the density variations in the X-ray images, and for each board based on the board scanner knot detection. This board scanner offers very high knot-detection precision thanks to the combination of a traditional vision system and laser-based tracheid measurements, which helps avoiding misclassification of dirt and stains as knots.

Matching of data

The knot fingerprints of all 22 926 logs were stored in a database. Each board fingerprint was then compared to all log fingerprints in the database in order to find the best matching log, and a reliability measure was calculated for each match. By setting a threshold on this reliability measure, it was possible to eliminate the most uncertain matches.

The share of boards being matched to logs was calculated using four different reliability thresholds. For each threshold, the share of correct matches was calculated by comparing the suggested best match to the true log identity in the manually matched data set.

RESULTS AND DISCUSSION

Matching results

Fig. 3 shows the matching of a board fingerprint to two different log fingerprints in the database. For most fingerprints, the matching was unambiguous; for Scots pine it was possible to trace 94% out of all boards to the correct log, and for Norway spruce this number was 93%. When using a threshold on the reliability measure, it was possible to reach 99% matching correctness while still tracing around 89% of the pine boards and 87% of the spruce boards. The results for the four different thresholds are shown in Fig. 4.

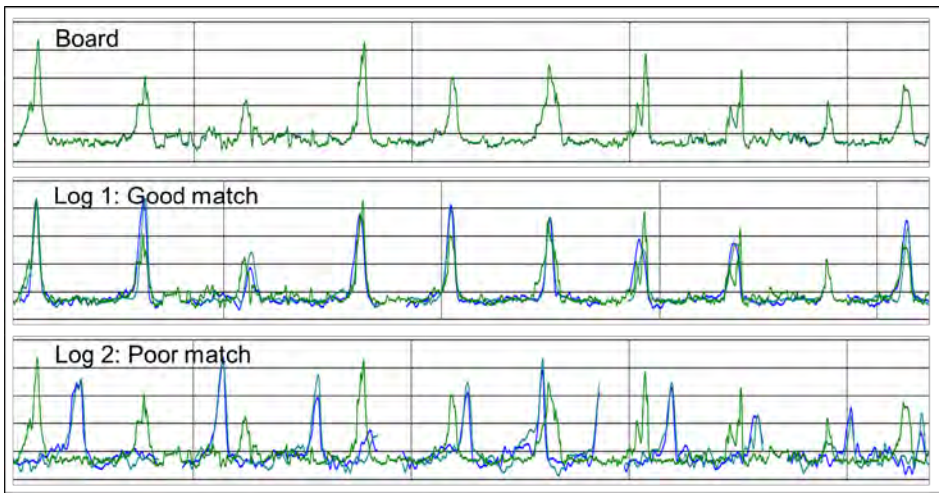

Fig. 3.

Matching of a board fingerprint to two different log fingerprints, log 1 being the correct log.

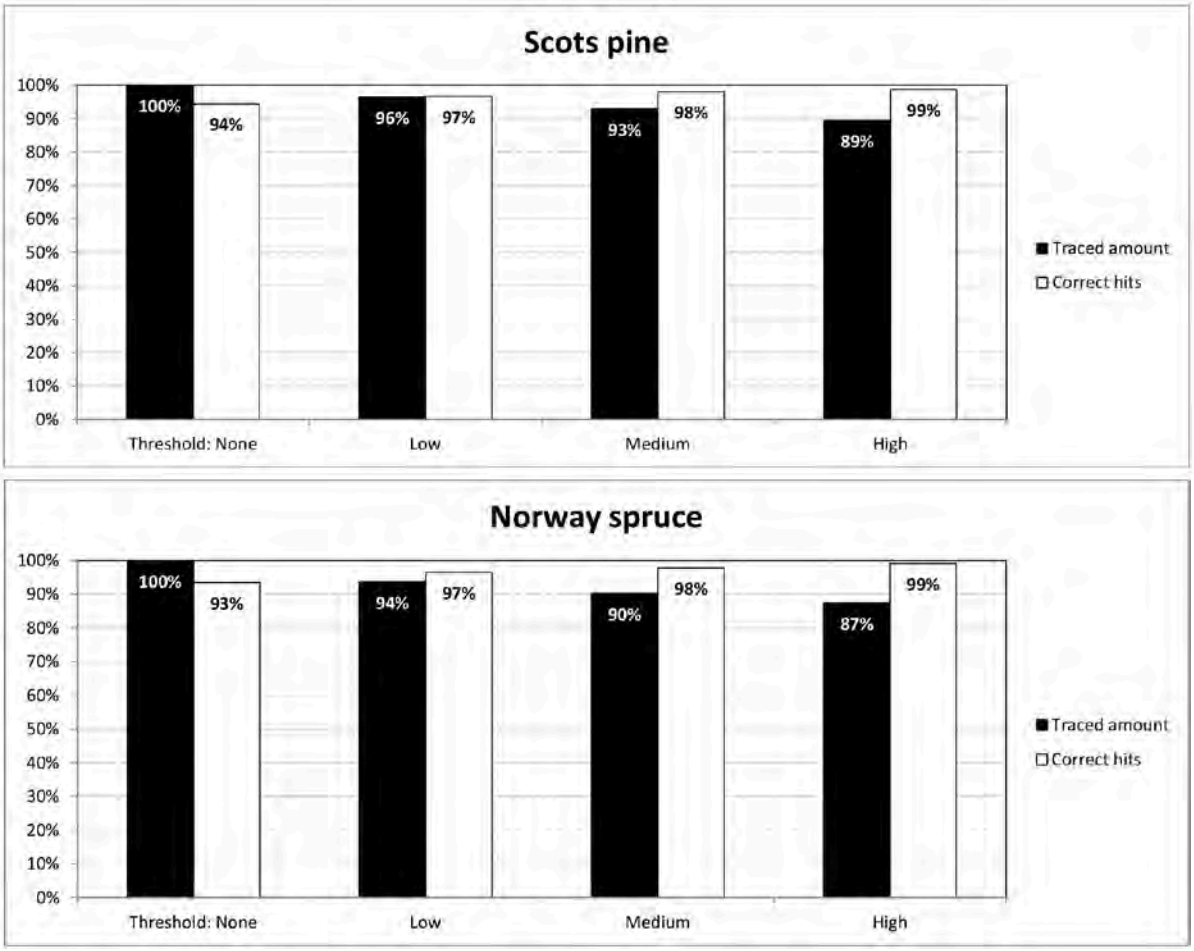

Fig. 4.
The share of Scots pine and Norway spruce boards being traced to logs (black) and the share of correctly traced boards (white) for different thresholds of the reliability measure.

Future Research on the Digital Sawmill

The traceability method described above was found very successful and the host sawmill decided to invest in an RS-BoardScannerQ of their own, which was installed beginning of 2017. This will enable the accurate and noninvasive tracing of almost every log to boards, thereby allowing the collection of big data describing the sawmill process. This data connects the properties of the raw material and the final products, thus offering completely new opportunities to adapt the sawmill production both to customer demands and to actual raw material properties.

A new research project called "The Digital Sawmill" has also been launched, aiming at implementing a more fine grained traceability along the whole process chain, connecting data from all relevant systems in addition to the X-ray log scanner and the board scanner data described here (Fig. 5). The project will investigate how this kind of big data can be used for production optimization and control. Some of the possibilities that will be investigated are:

1. What kind of logs should be used for each product?
2. How should the logs be sorted and handled in order to achieve maximum value of sawn goods?
3. What are the real costs associated with each product?
4. Are there some logs that generate a negative value and should never enter the sawmill?
5. Can X-ray log scanner data be used for non-contact strength grading of boards?
6. How can X-ray data be used for improved decision making in the wood drying process, preserving energy while obtaining better results?
7. Could collected production data be used by industrial customers in further processing plants?
8. The use of connected data for process monitoring: In a sawmill, there are many machine settings that can go wrong and if the process is not being properly

monitored, any hard-earned gains from process optimization is quickly being lost. On-line access to connected production data could allow the mill to quickly detect deviations from expected behavior, thereby avoiding expensive losses in volume or quality of sawn goods.

In short, with access to full information about the relation between raw materials and final products, a world of new possibilities opens up! In the digital sawmill, production control will be adaptive and able to quickly adjust sorting, sawing, drying and trimming to fluctuations both in market demands and incoming raw material properties.

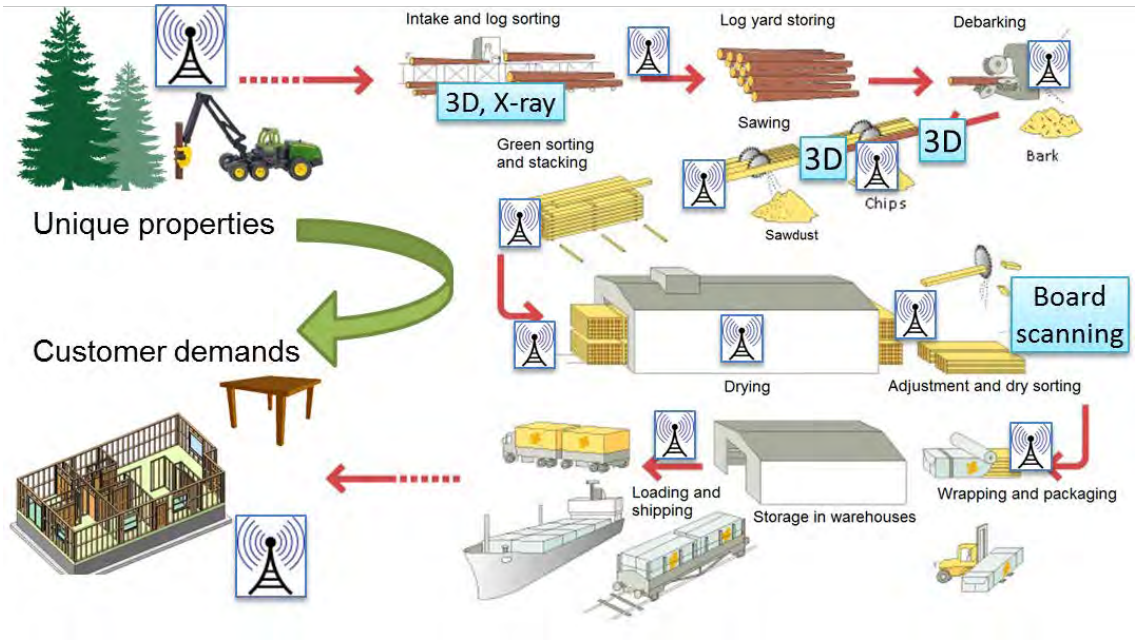

Fig. 5.
The digital sawmill where production data are collected and traced on an individual basis through the whole production chain. The collected big data allows for advanced production control and gives understaning how the unique properties of the raw material are best utilised in order to meet the customer demands on the final products. (Adapted from an image by the Swedish Forest Industries Federation).

CONCLUSIONS

Fingerprint traceability has been proven successful for automatically and accurately identifying which log the sawn boards originate from, for both Scots pine and Norway spruce. It was found that 99% correct matches could be reached while tracing 87-89% of all boards.

Connected production data will provide new information on the relation between raw material properties and final product properties. This information will enable the implementation of an adaptive production control of sorting, sawing, drying and trimming, in order to best meet market demands.

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MECHANIZATION OF WOODEN FRAMED LAMELLAR PANELS DESIGNED FOR PACKAGING CRATES, WAY OF INCREASING PRODUCTIVITY AND QUALITY

Adriana FOTIN
Lecturer dr.eng.- Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universității nr. 1, 500068 Brasov, Romania
E-mail: adrianafotin@unitbv.ro

Ivan CISMARU
Prof.dr.eng. – Transilvania University of Brașov, Member of the Academy of Romanian Scientists
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universității nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: icismaru@unitbv.ro

Abstract

Wood packaging crates having generally a single cycle of use must be cheap in order not to affect the price of the products it stores or transports. For this reason, manufacturing costs (materials, energy consumption and labour) should place the packaging cost at the maximum 5% of the price of the product stored or transported, or even less. For this reason, all the ideas related to manufacturing process that could affect the cost of packaging must be taken into account. A significant amount of labour is consumed in forming panels in the structure of the boxes which involves the consolidation of lamellas (piece by piece) with nails or staples on the reinforcement elements (wooden cross rails or frames).The paper aims to present technological improvements (with a different degree of mechanization) of the lamellar panels' industrial flow, which may decrease the labour consumption on the product and implicitly may influence the price of the product. These improvements may help the activity of this industrial sector to be profitable in valorisation the poor quality and small sized wood.

Key words: *frames; crates; lamellas; packaging.*

INTRODUCTION

The woodworking industry in the manufacturing processes produces wastes, such as small pieces of wood unusable in the furniture manufacturing or for doors and windows or other products used for buildings. On the other hand, inferior quality wood due to the natural defects of wood, such as logs, edges, laths, are generally used in the parquet flooring industry and in the wood packaging industry. Thus, wooden packaging crates or containers used for storing and transporting products of various origins, such as agricultural, food or industrial ones are the final products of the low quality wood.

The efficiency and maintenance of the wood packaging industry is requested, because of the high volume of wood that could not be utilized in the other (basic) sectors of the wood industry. More than that, starting from the idea that packaging does not have to "load" the price of stored or transported products, the wooden packaging (crate type) would be among the few that would fulfil this role.

If it is also considered that at the end of the lifetime the packaging disposal must not affect the environment, it can be said again that wooden packaging can fulfil this condition, being burned (resulting in thermal energy usable in diverse utility thermal systems) or resulting in compost by biodegradation process.

As shown in a previous paper (Cismaru and Fotin 2016), wood packaging does not transfer dyestuffs or toxic products to stored goods during use. Under these circumstances, if wood packaging products are collected and recycled, they may be used in several exploitation cycles. Thus, just a small share value is transferred to the products and the efficiency of capitalizing these types of products significantly increases.

However, the cost of production remains the basic element that must be controlled, thereby ensuring the profitability of the packaging sector, which is of great importance to the general economy and to the woodworking industry in particular.

Meanwhile, there have been industries that have tried to take over this area of packaging (glass, plastics, metals, textiles, cardboards), some developing large-scale activity (plastics and cardboards) others quickly quitting as unprofitable (metals, glass).

Plastics, because of the flexibility of the technological process, have gained massive positions in the crate-type and containers packaging. The disadvantages are their higher prices and problems in terms of recycling or ecological disposal at the end of the lifetime.

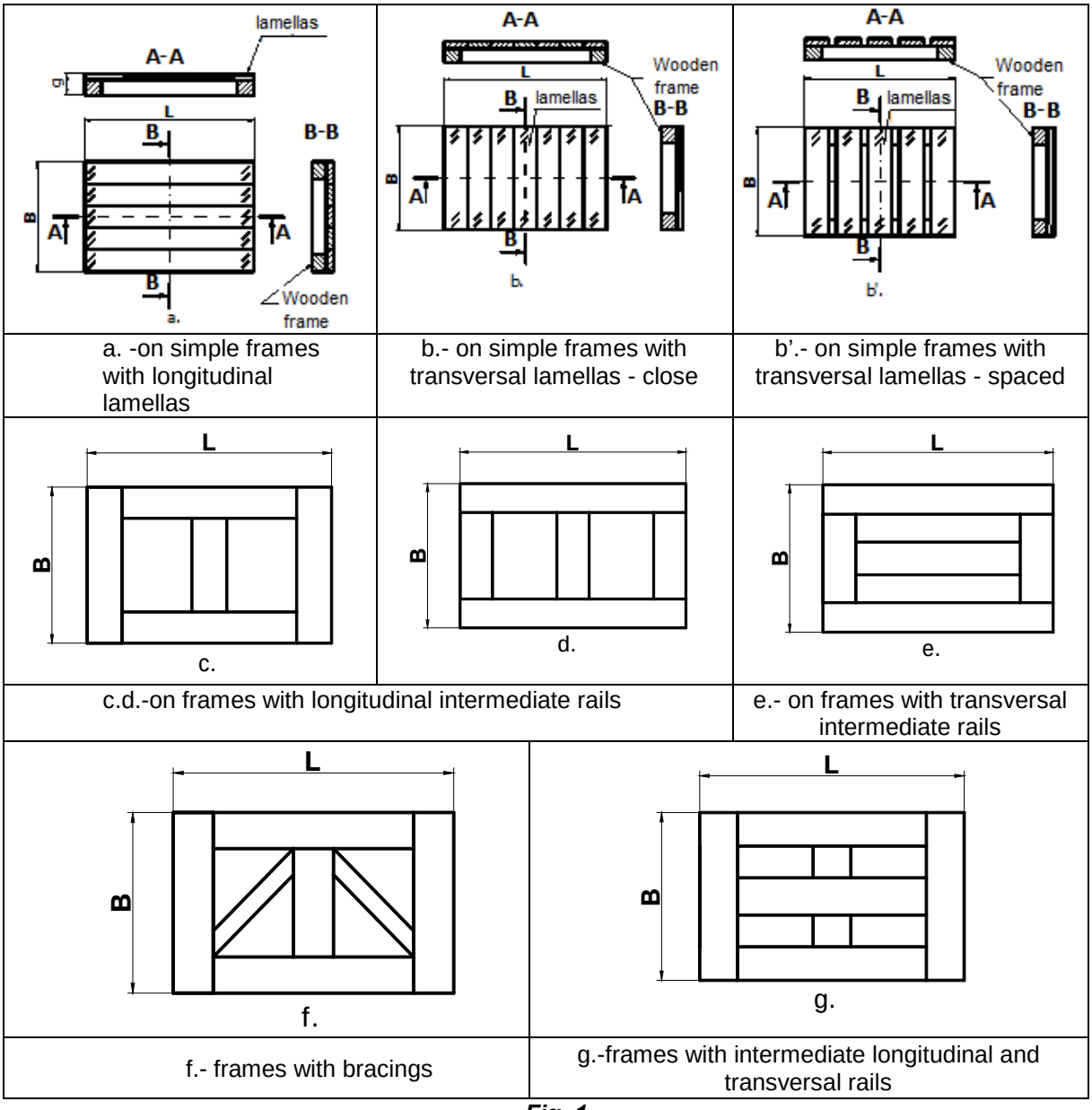
Plastics used at a large scale for all kind of products are nowadays limited in use because of their high resistance to biodegradation, and because combustion recycling creates many hazardous and toxic gases.

STRUCTURE OF WOODEN PACKAGING CRATE

Wooden crates are made of various shapes and structures, depending on the transported goods, their stacking mode (horizontally or vertically), the structure of merchandise (granular, in bulk or packed, etc.).

The wooden packaging crates are designed depending on the goods to be transported, the way of storage in the warehouses or in the means of transport, resulting in the following input data:

- inner volumes (implicitly the interior dimensions) of the crates;
- consolidation solutions (cross rails or frames);
- solutions of orientation of the lamellas against the other structural components (cross rails, frames), being positioned longitudinally or transversally, as the case may be;
- additional consolidation solutions using diagonal rails, longitudinal or intermediate rails;



a. -on simple frames with longitudinal lamellas

b.- on simple frames with transversal lamellas - close

b'.- on simple frames with transversal lamellas - spaced

c.d.-on frames with longitudinal intermediate rails

e.- on frames with transversal intermediate rails

f.- frames with bracings

g.-frames with intermediate longitudinal and transversal rails

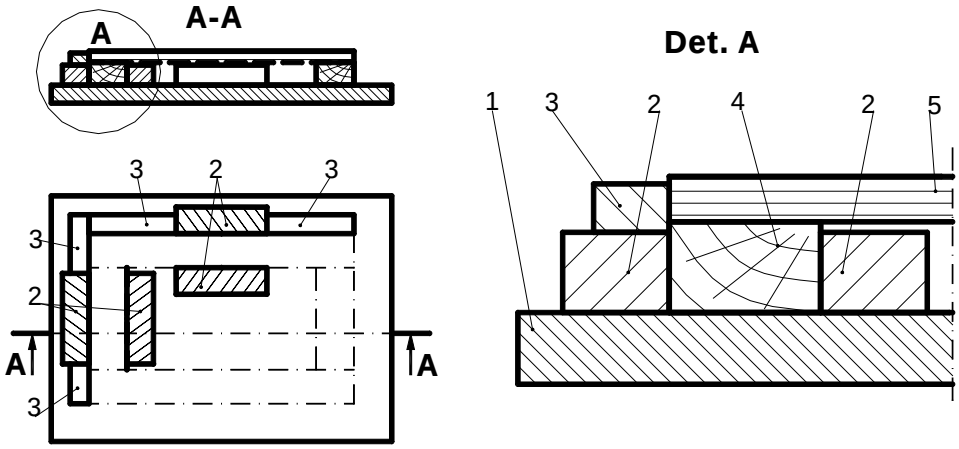
Fig. 1.
Wooden framed panel types for packaging wooden crates (L>B>g).

The following results after calculation are obtained:
- the dimensions (lengths, widths and thicknesses) of the lamellas (Cismaru 2016, Cismaru and Fotin 2012);
- the dimensions of the reinforcing elements (cross rails) and their orientation in the product (vertical or horizontal) (Fotin 2012);
- dimensions and number of reinforcement accessories (staples, nails, screws);
- consolidation solutions with accessories, in the idea that the crates are fixed or foldable, etc.

PANELLING THE FRAME

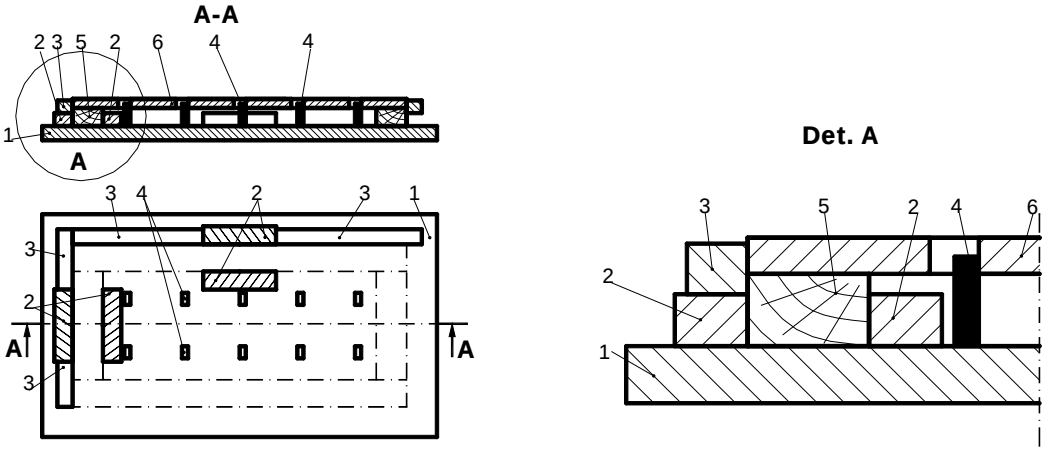
Attaching the lamellas to the frames in the panelling phase involves connecting the structural components with nails, staples or screws so that the accessories do not come in contact with the transported goods and prevent them from scratching or influencing the good quality of the products.

In order to fix the lamellas to the frames, they will be placed in well defined, close or spaced positions, identical for all batches. Specialized technological devices are used for close lamellas (Fig. 2) or for spaced ones (Fig.3).



1.-device plate; 2.-guiding supports for the frame positioning on the device ; 3.-end support of the close lamellas relative to frame end; 4.-frame; 5.-lamellas

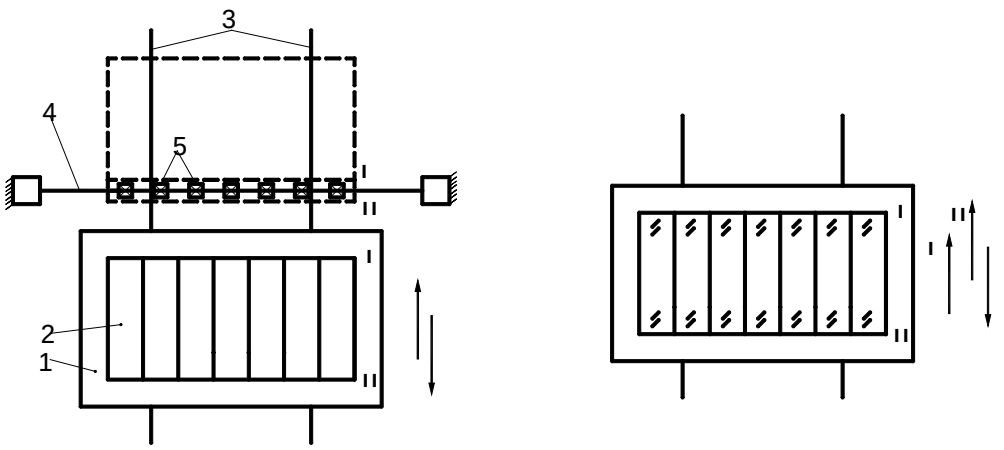
Fig. 2.
Technological device designed to position the close lamellas for their consolidation on the frame.



1.-device plate; 2.-guiding supports for frames; 3.-end support of the spaced lamellas relative to frame end; 4.-supports equally spaced for placing the lamellas; 5.-frame; 6-lamellas

Fig. 3.
Technological device designed to position the spaced lamellas for their consolidation on the frame.

Consolidating the frames by attaching the lamellas to the frames by means of the accessories is done manually or by moving the device in an installation to bring the lamellas under the beam on which the pneumatic or mechanical systems of stapling or nailing are located (see Fig. 4).



1.-Technological device situated on the machine table; 2.-frame and lamellas positioned close ore spaced; 3.- linear guides of the equipment for moving the mobile table; 4.- beam support, on which the nail or staple systems (mechanically or pneumatically) are attached; 5.-stapling or nailing systems.

Fig. 4.
Scheme for equipment of applying reinforcement accessories (nails or staples).

Beginning with the scheme in Fig. 4, one can think of the transition from the semi-mechanical or manual system to a mechanized system, respecting the operations and the technological phases in their sequence, the manipulations of the structural elements being executed mechanically, as shown in Figs. 5 and 6, for both close and spaced lamellas.

The equipment shown in Figs. 5 and 6 may automatically execute the wooden panels of the crates with continuous feed speed (variant A) or intermittent feed speed (variant B). The result is a high level of productivity simultaneously with the relieving of the operators by a relative high physical effort for nailing or stapling.

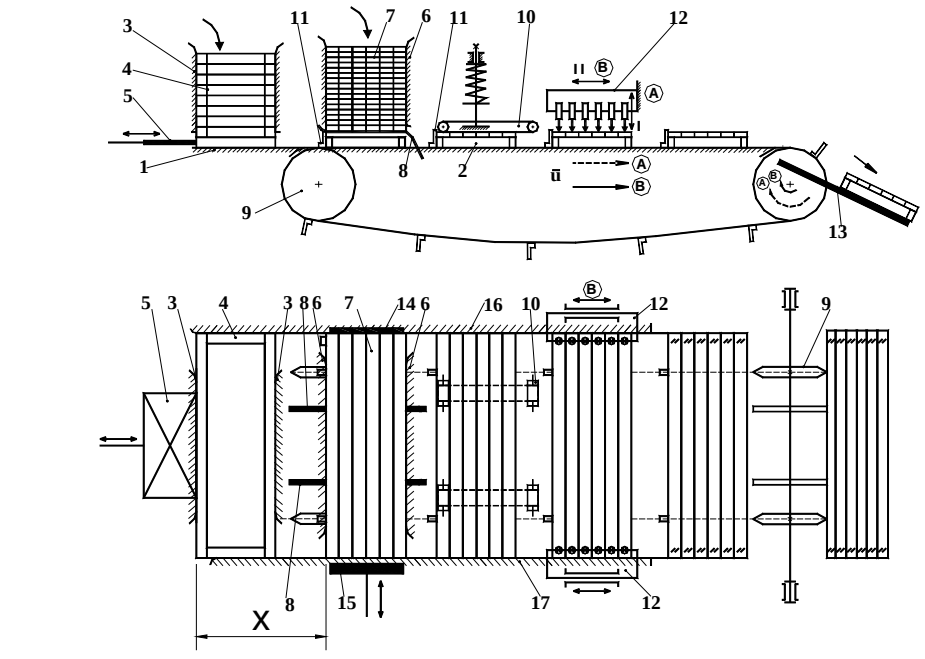


Fig. 5.
Equipment of panelling the frames with close or spaced lamellas.

The operation of the equipment presented in Fig. 5 requires the use of a cyclic (alternatively) pusher 5, which feeds the frame system 4, stored in a gravitational bunker. The frames are pushed under the bunker 6 in which the lamellas 3 are deposited in layers including one panel structure. The lamellas are supported on two bars 8 which place them above the frames pushed with the system 5. The frames are taken over by the conveyors 9 with the help of the claws and in the same time the layer of the lamellas from the bunker 6 are deposited on the frame. The conveyors 10 (not driven and applying a braking force on the lamellas) are used to set an adequate positioning of the lamellas. After positioning the lamellas at the ends (with the help of supports 14 and of a vibrating plate 15), they are placed correctly on the frame according to their width. After forming the panel, it reaches the nailing or stapling system 12, which pneumatically or mechanically applies the nails or staples (on the position - variant A, or by simultaneous advance with the panel and return - variant B).

The system 12 has an equal (or double) number of nailing or stapling systems or double-headed devices mounted on the beams.

After applying the nails or staples, the panel is transferred through a sloping plane to the storage location.

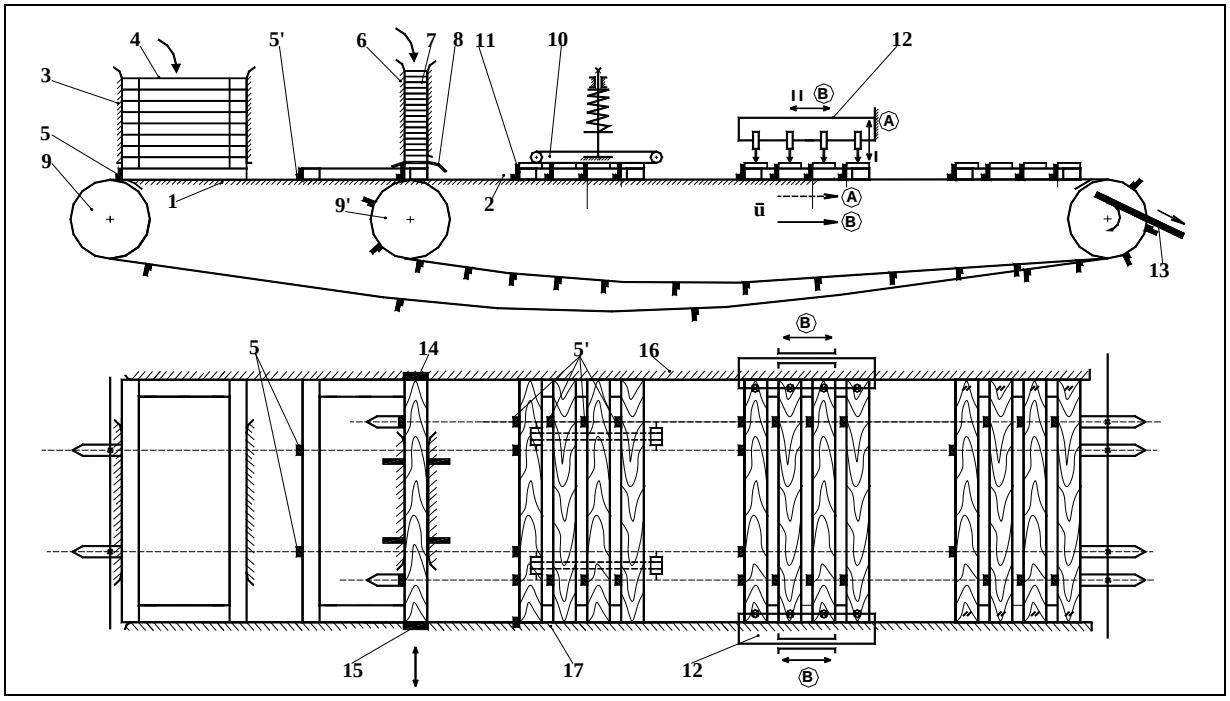


Fig. 6.

Equipment for forming wooden framed panels for crates by using frames and spaced lamellas.

Movement I is the one that performs the application of the staples or nails (application by the intermittent feed speed of the frames), and the movement II is that of advancing and returning - to the continuous feed speed application of the frames.

The components of the installation are as follows:

1. Support of the frames stock;
2. Supports for framed panels when passing through the installation;
3. Frames storage bunker;
4. Stock of frames;
5. Alternative frame pusher (variant A);

The claws of the conveyor 9, which drive the frames (Fig.5) or frames and lamellas (Fig. 6);

5'. Claws of the conveyor 9 '(Fig. 6), which drive each lamella;

6. Lamellas storage bunker;
7. Stock of lamellas;
8. Positioning beams of the last lamella layer;
9, 9'. Crawler conveyors with claws;
10. Non-driven conveyors - acting as brakes;
11. Drive claws;
12. Nailing or stapling system;
13. Sloping evacuation plan;

14. End panel - for laying the lamellas in the same plane;
15. Vibrating panel having the role of keeping the lamellas on the same plane;
16, 17. Side guidance of the frames and lamellas (left-right).

The correct operation of the equipment involves the synchronization between the movements and maintaining the position of the lamellas on the frames until the nails or staples are applied.

In the case shown in Fig. 6, the two conveyors 9 and 9 'must have the same peripheral velocity and a part of the claws located on the same plane (those which drive the frame and the last lamella on the frame). Similarly, the claws that ensure the spacing of the lamellas on the frame (5 ') must be placed within the same plane.

CONCLUSIONS

Mechanization of the wood packaging crates manufacturing sector, no matter if they are used as packaging for agricultural, food or industrial products can be considered as one of the main ways to control production costs and the quality of these products, in the conditions when there is so much interest in greening the recycling of products that have exceeded their lifetime.

Wood and cardboard, as well as lignocellulosic composites could be the solution for the raw materials used for packaging, so to massively solve the packaging recycling.

More than that, if we also add the advantage that wood is a natural renewable product, we could say that the use of wood, cardboard or lignocellulosic composites as packaging materials will not have the effect of consuming the Earth's reserves. In fact, the wood grows constantly and people do very little for tree growth.

Since wood is also an inexpensive material, the cost of the packaging is expected to be in the acceptable percentage rate with the stored or transported products.

To design packaging using all kinds of raw materials such as solid wood, veneers, paper, cardboard or other lignocellulosic composites is particularly necessary and could be achieved by a more active marketing activity in the packaging area in the direction of its design and technology.

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STUDY REGARDING THE INFLUENCE OF THE TOOL GEOMETRY AND FEED RATE ON THE DRILLING QUALITY OF MDF PANELS

Mihai ISPAS
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: ispas.m@unitbv.ro

Sergiu RACASAN
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: sergiu.racasan@yahoo.com

Abstract

Wood based panel such as medium density fibreboards (MDF)are extensively used in the furniture industry. The most common machining process used for joining is drilling. In order to achieve a good surface quality and minimize the delamination, two of the main parameters must be taken into consideration while drilling: the tip angle of the drill bit and the feed speed. The objective of this study is to analyze the influence of the tip angle of the drill bit and the feed speed on the processing quality evaluated by the size of delaminations at the entrance side and the exit side of the drill bit for two types of drills: flat and twist (helical) drills. To assess the defect, a non-dimensional parameter was used: the delamination factor. The results showed that, in general, the combination of small tip angle with low feed rate minimizes the delamination at drilling.

Key words: *drilling; delamination; MDF.*

INTRODUCTION

Drilling is one of the most usual and frequent type of processing in the wood industry with the most common application in the furniture industry. This has developed along years into a massive production of furniture, where one of the main raw material is fibreboard, mainly medium density fibreboard (according to FAOSTAT, the worldwide fibreboard production in 2016 was of app. 119 mil.m3).

The operation of free drilling, which is common for on-line processes, raises a surface defect noticed, at the medium density fibreboards, to occur around the drilled holes. Delamination is a processing defect which consists of a local detachment of the coating layer engaging chips/particles pull-offs from the fibreboard surface. This phenomenon can occur during drilling at the entrance side as well as at the exit side (for drilled through holes). Its magnitude depends on the processing parameters and can be used as an indicator of the drilling quality (Davim et al. 2008).

Some long ago, Radu (1967), in his extensive study on drilling with twist drills, referred to the quality of drilling the particleboards in terms of visual qualifications of the surface in the neighborhood of the processed holes. Parameters, as tool feed speed and tool geometry, were amongst the ones investigated, but the qualifications were limited to subjective qualitative assessments as: "good", "weak", "slight increase", "slight decrease".

More recently, the delamination caused by drilling the wood based panels, especially medium density fiberboards (MDF), was quantitatively assessed by using a parameter called delamination factor, Fd. Hence, Davim et al. (2008) investigated the relationships and parametric interaction between the feed rate and the cutting speed on the Fd at entry and exit side of the holes in drilling the MDF. Two types of MDF panels, melamine coated and veneered, were tested using cemented carbide (K20) drills. The Fd decreased with the increase of the cutting speed and increased with the feed rate for both materials. Palanikumar et al. (2009), Prakash et al. (2009) studied the performance characteristics given by Fd in drilling operations of MDF boards using carbide tools. The machining parameters considered were: the spindle speed, the feed rate and the drill diameter. They found that Fd decreases with the increase of the cutting speed and increases with the feed rate and drill diameter. Prakash and Palanikumar (2011) investigated the influence, at MDF, of the spindle speed, feed rate and drill diameter on the surface roughness of the processed hole. The experimental result revealed that the most significant drilling parameter for the surface roughness was the feed rate followed by the cutting speed. Valarmathi et al. (2013) analyzed the influence of cutting parameters in a systematic approach on delamination in drilling of prelaminated MDF wood panels with

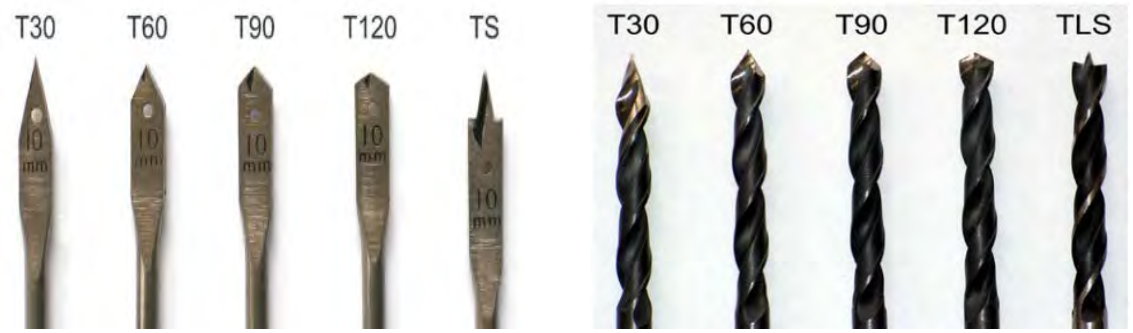
High Speed Steel (HSS) twist drills of different diameters. The results showed that the optimal conditions for minimizing the delamination are high spindle speed, low feed rate, and small drill diameter (6 mm).

OBJECTIVE

The objective of this study is to evaluate the influence of the tool type, geometry and feed speed on the drilling quality of the medium density fibreboards (MDF) panels. The processing quality was evaluated by de size of delamination measured, both, at the entrance and exit sides of the drilled holes. In order to evaluate the delamination damage occurred during drilling, the delamination factor was taken into consideration.

MATERIAL, METHOD, EQUIPMENT

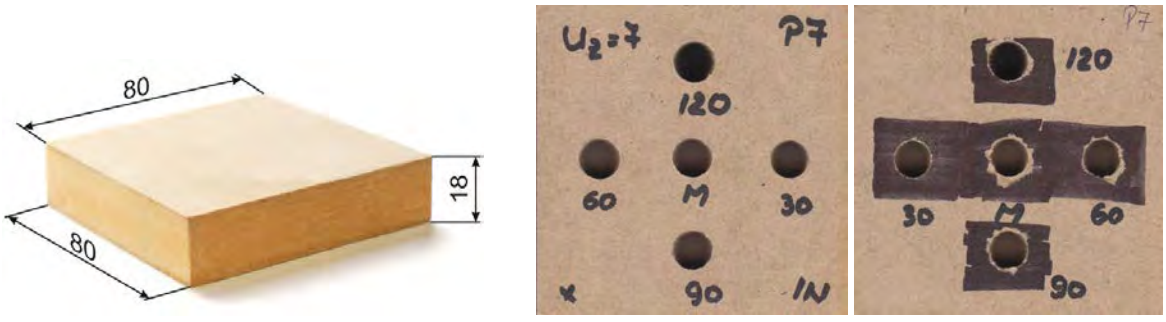
The experiments were performed using 4 flat drill bits (rake angle γ = 0°) with 10mm cutting diameter, with different tip angles ($2\kappa_r$ = 30°, 60°, 90°, 120°) and one spade drill bit (Fig. 1a). The clearance angle of all drills was the same α = 20°. The symbols used for these drills were tip angle related: T30, T60, T90, T120, respectively TS for the spade drill. The second type of drills used were 4 twist (helical) drill bits with 10mm cutting diameter, with different tip angles ($2\kappa_r$ = 30°, 60°, 90°, 120°) and one lip and spur drill bit (Fig. 1b). The clearance angle of all drills was the same α = 20°. The symbols used for these drills were tip angle related: T30, T60, T90, T120, respectively TLS for the lip and spur drill.



a. b.

Fig. 1.
Types of drills used for processing: a – four flat drills with the tip angle $2\kappa_r$ of 30°, 60°, 90°, 120° and one spade drill; b – four helical drills with the tip angle $2\kappa_r$ of 30°, 60°, 90°, 120° and one lip and spur drill.

For both types of drills, a set of fourty square samples □80mm were cut from a single pre-laminated 18mm thick particleboard (Fig. 2a).



a. b.

Fig. 2.
Samples used for drilling: a – sample dimensions; b - MDF specimens drilled with the flat drills (T30, T60, T90, T120, TS) when the tooth bite was 0.7 mm at the entrance side and the exit side.

They were divided into four groups of ten specimens each. Each specimen was drilled with five different drills (T30, T60, T90, T120, TS respectively, T30, T60, T90, T120, TLS) (Fig. 2b).

Each group of ten specimens was drilled with a different feed speed so that the tooth bite, f_z, was different, having the following values: 0.1, 0.3, 0.5 and 0.7mm. The rotation speed of the drills was the same, n = 3000rpm. This led to four feed speed values, vf = 0.6, 1.8, 3.0 and 4.2m/min.

The processing machine was a CNC processing centre type ISEL GFV/GFY, which allowed the exact set-up of the drills rotation speed and of the feed speeds (Fig. 3).

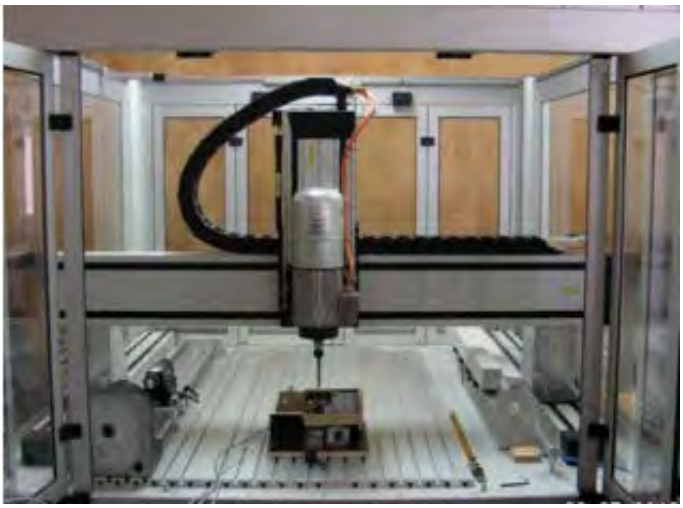


Fig. 3.
CNC processing centre type ISEL GFV/GFY used for drilling.

After drilling, each hole diameter was measured with an electronic calliper, with a 0.01mm precision, on two perpendicular directions and a mean diameter was calculated for both hole sides (entrance and exit), as can be seen in Fig.4. All drilled specimens were then scanned on both sides and received codes, IN, for entrance side, respectively OUT, for exit side (Fig. 2b).

The scanned images were used to evaluate the delaminations that occurred around each hole, on both sides. The delamination was evaluated by the delamination factor F_d , given in equation 1,

$$F_d = \frac{D_{\max}}{D} \tag{1}$$

- where D_{max} is the diameter of the circle circumscribed to the defect, while D is the mean hole diameter given by calliper measurements D1 and D2 (Fig. 4)

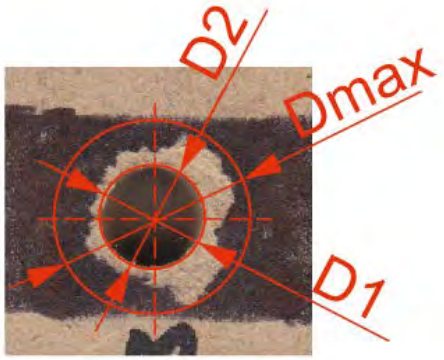


Fig. 4.
The measurement of diameters for calculation of the delamination factor in order to evaluate the delamination caused by drilling on the MDF sample.

RESULTS AND DISCUSSION

The mean values, standard deviation and coefficients of variation were calculated for the delamination factor F_d (Table 1 and Table 2, for flat drills).

Table 1

The mean values, standard deviations and coefficients of variation for the DELAMINATION FACTOR, at the ENTRANCE SIDE of the specimens drilled with FLAT DRILLS, for various feed rates and drill geometries.

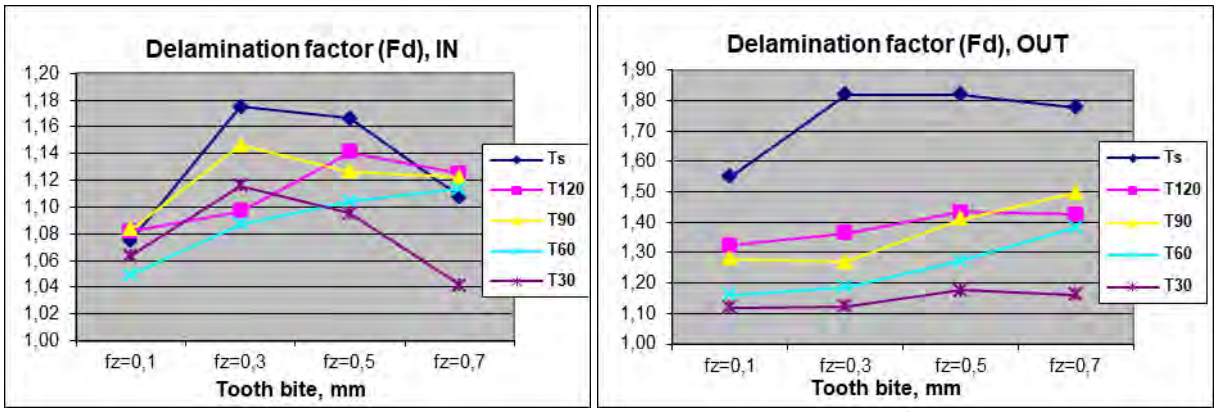
IN	fz = 0,1			fz = 0,3			fz = 0,5			fz = 0,7		
	mean	SD	cvar (%)	mean	SD	cvar (%)	mean	SD	cvar (%)	mean	SD	cvar (%)
T30	1,06	0,05	4,98	1,12	0,06	5,34	1,10	0,05	4,81	1,04	0,02	2,33
T60	1,05	0,01	1,17	1,09	0,03	2,35	1,10	0,03	2,93	1,11	0,04	3,29
T90	1,08	0,01	0,85	1,15	0,04	3,74	1,13	0,04	3,52	1,12	0,04	3,33
T120	1,08	0,04	3,72	1,10	0,02	2,16	1,14	0,04	3,74	1,12	0,04	3,22
TS	1,08	0,04	3,48	1,18	0,05	3,99	1,17	0,07	5,65	1,11	0,07	6,04

Table 2

The mean values, standard deviations and coefficients of variation for the DELAMINATION FACTOR, at the EXIT SIDE of the specimens drilled with FLAT DRILLS, for various feed rates and drill geometries.

OUT	fz = 0,1			fz = 0,3			fz = 0,5			fz = 0,7		
	mean	SD	cvar (%)	mean	SD	cvar (%)	mean	SD	cvar (%)	mean	SD	cvar (%)
T30	1,12	0,04	3,64	1,12	0,04	3,29	1,18	0,07	5,60	1,16	0,03	2,84
T60	1,16	0,03	2,16	1,19	0,04	3,70	1,27	0,08	5,96	1,38	0,14	9,99
T90	1,28	0,05	4,26	1,27	0,05	4,17	1,41	0,18	12,80	1,50	0,19	12,58
T120	1,32	0,12	8,84	1,36	0,10	7,35	1,43	0,14	9,59	1,42	0,12	8,35
TS	1,55	0,29	18,64	1,82	0,18	9,76	1,82	0,15	8,17	1,78	0,12	7,03

By drilling with flat drills, there was a general trend of delamination increase with the increase of the tooth bite (feed rate) (Fig. 5).



a. b.

Fig. 5.

The variation of the delamination factor with the tool feed speed and geometry, using flat drills (figures represent mean values): a - at the entrance side of the drilled specimens; b - at the exit side of the drilled specimens.

Whatever the specimen (hole) side, the biggest delaminations occured when drilling with the spade drill bit (TS), regardless of the used feed speed (exception: delaminations on entrance side produced when drilling with f_z=0.1mm and f_z=0.7mm). This trend can be noticed better on the exit side of the specimens. On the entrance side, where the delamination is very small (sometimes almost non-existent) and the maximum value of the delamination factor is F_d=1.17, the influence of the tool geometry on the delamination size did not have a clear trend.

As expected the delamination factor has higher values on the exit side of the specimens than on the entrance side.

The mean values, standard deviation and coefficients of variation were also calculated for the delamination factor F_d, by drilling with the twist drills (Table 3 and Table 4).

Table 3

The mean values, standard deviations and coefficients of variation for the DELAMINATION FACTOR, at the ENTRANCE SIDE of the specimens drilled with TWIST DRILLS, for various feed rates and drill geometries.

IN	fz = 0,1			fz = 0,3			fz = 0,5			fz = 0,7		
	mean	SD	cvar (%)	mean	SD	cvar (%)	mean	SD	cvar (%)	mean	SD	cvar (%)
T30	1,00	0,00	0,00	1,01	0,02	2,18	1,02	0,04	3,48	1,10	0,04	3,95
T60	1,00	0,00	0,00	1,01	0,03	2,60	1,06	0,06	6,01	1,17	0,08	7,10
T90	1,00	0,00	0,00	1,01	0,03	3,14	1,05	0,06	5,66	1,16	0,05	4,23
T120	1,00	0,00	0,00	1,01	0,02	2,44	1,12	0,06	5,03	1,20	0,07	5,73
TS	1,00	0,00	0,00	1,02	0,04	3,49	1,02	0,04	4,25	1,12	0,06	5,26

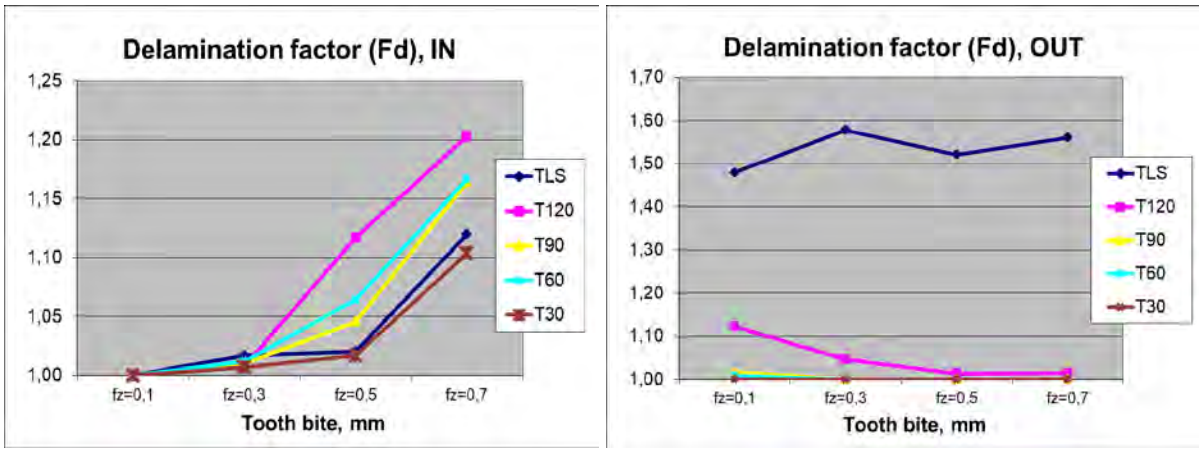
Table 4

The mean values, standard deviations and coefficients of variation for the DELAMINATION FACTOR, at the EXIT SIDE of the specimens drilled with TWIST DRILLS, for various feed rates and drill geometries.

OUT	fz = 0,1			fz = 0,3			fz = 0,5			fz = 0,7		
	mean	SD	cvar (%)	mean	SD	cvar (%)	mean	SD	cvar (%)	mean	SD	cvar (%)
T30	1,00	0,00	0,00	1,00	0,00	0,00	1,00	0,00	0,00	1,00	0,00	0,00
T60	1,01	0,03	2,55	1,00	0,00	0,00	1,00	0,00	0,00	1,00	0,00	0,00
T90	1,02	0,05	5,13	1,00	0,00	0,00	1,00	0,00	0,00	1,00	0,00	0,00
T120	1,12	0,08	7,17	1,05	0,05	5,22	1,01	0,04	4,09	1,01	0,04	4,33
TS	1,48	0,18	12,03	1,58	0,23	14,82	1,52	0,10	6,50	1,56	0,14	9,07

The samples drilled with the twist drills have small delaminations on the entrance side, for drilling with a feed rate f_z=0.7mm for all types of twist drill bit (with very low values), while, for the other feed rates (f_z=0.1mm, f_z=0.3mm and sometimes f_z=0.5mm) the quality of the processing was very good (Fig. 7).

On the exit side of the specimens, drilling with T30, T60, and T90 twist drill bits, gave a very good quality also, without delaminations, while drilling with T120 drill bit, delaminations appeared only at a feed rate of f_z=0.1 and f_z=0.3, with very low values for the delaminiation factor (Fig. 6).



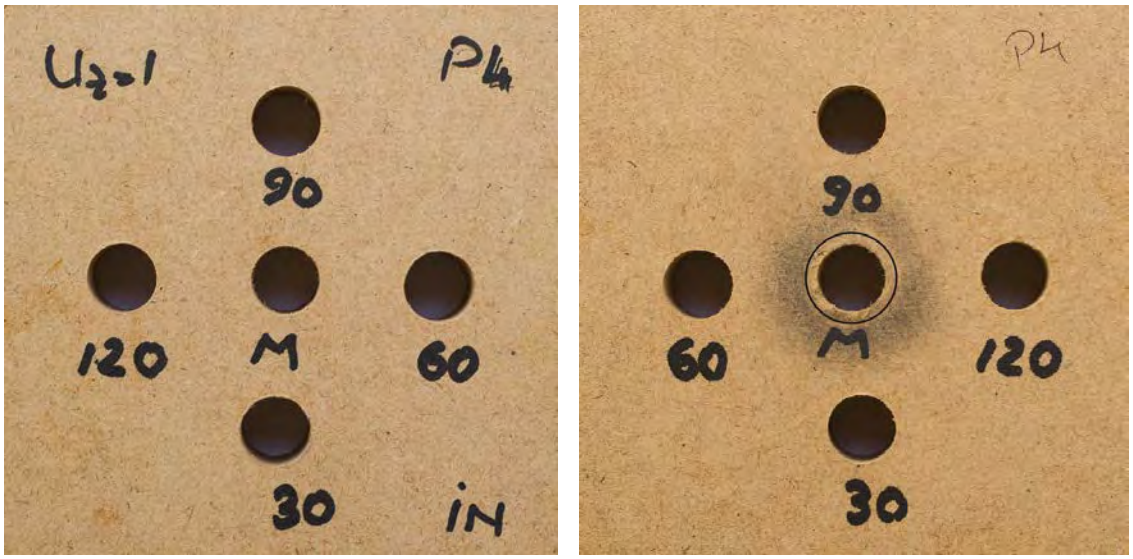
a. b.

Fig. 6.

The variation of the delamination factor with the tool feed speed and geometry, using twist drills (figures represent mean values): a - at the entrance side of the drilled specimens; b - at the exit side of the drilled specimens.

Whatever the specimen (hole), the biggest delaminations occured on the exit side, when drilling with the lip and spur drill bit (TLS), regardless of the used feed speed.

Drilling with the twist drill bits gave lower values for the delamination factor and a better quality of the processing, than drilling with the flat drill bits.



a. b.

Fig.7.
Sample drilled with twist drills and tooth byte f_z of 0.1 mm:
a - at the entrance side b - at the exit side.

In general, T30, T60 and sometimes T90 gave a better processing quality than T120. TLS gave the worse quality by far, on both types of drills: flat drills and twist drills. However, because of its elongated tip geometry, processing with T30 drill bit in case of thin boards is limited to manufacturing through holes rather than with limited depth (Fig. 8).

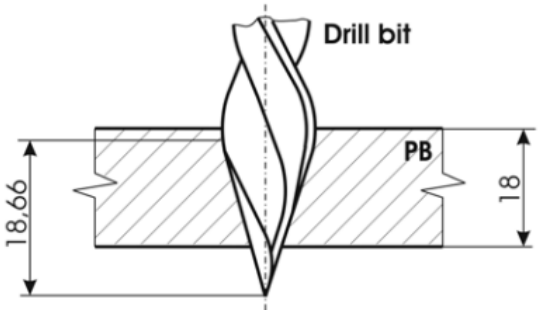


Fig. 8.
Limitation of T30 drill bit and its elongated tip geometry when manufacturing thin boards.

CONCLUSIONS

Free drilling is a common operation for medium density fibreboards in the furniture industry. The quality of this operation can be assessed by the delamination occurring around the hole. This paper examined this defect by means of a non-dimensional parameter one used also by other researchers, the delamination factor. The influence of the feed rate (tooth bite) and tool geometry (tip angle) was assessed by the above quality parameter.

Generally, the delamination increased with the increase of the tooth bite (feed rate) for all drill types and geometries. The defect zone was larger at the exit side of the drill compared to the entrance side with the greatest amount for the spade drill, followed by the other drills (respectively for the lip and spur drill).

Whatever the specimen, drilling with the twist drill bits gave lower values for the delamination factor and a better quality of the processing, than drilling with the flat drill bits.

If delamination and flexibility of hole depth is considered, a flat drill with 60° tip angle gave the best quality for small feed rates. The spade drill and the flat drill with the greatest tip angle, 120°, do not seem appropriate for processing medium density fibreboards (MDF).This conclusion also applies in case of drilling with twist drills and lip and spur drill.

Further studies may complete these results for various rotation speeds and other types of drills to optimise the process quality at drilling pre-laminated particleboards.

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SURFACE ROUGHNESS OF BEECH (*Fagus sylvatica*) AFTER ACTION OF A CO_2 LASER BEAM AT DIFFERENT POWER OUTPUTS AND SCANNING SPEEDS

Lidia GURAU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: lidiagurau@unitbv.ro

Adrian PETRU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: adrianpetru_ro@yahoo.com

Abstract

The literature provides very little information about engraving or decorating wood using a laser beam. No study was found that considers the surface roughness of wood after such treatments. This paper therefore aimed to find the influence of varying the laser power output and scanning speed of a CO_2 laser beam on the surface roughness of beech wood (Fagus sylvatica) for aesthetic applications such as decorative drawing. Laser power outputs from 5.6 to 6.8W were tested in combination with scanning speeds from 100 to 500mm/s. The surface roughness was assessed with a robust filter and by following measuring and evaluation recommendations from previous research to reduce the bias from the wood anatomy. The surface roughness measured by a series of roughness parameters (Ra, Rq, Rt, Rk, Rpk, Rvk) increased with laser power and decreased with scanning speed.

Key words: *laser power; laser scanning speed; wood surface roughness; robust filtering; roughness parameters.*

INTRODUCTION

Laser beam technology has multiple applications in almost all known materials, but it has been extensively researched only for the metal industry.

A review of the application of CO_2 laser beams as a cutting tool for various materials was made by Radovanovic and Madic (2011), who also mentioned the application of a laser beam on cutting MDF, citing the work of Lum *et al*. (2000).

Laser engraving is the practice of using lasers to engrave or mark an object. Laser engraving is the removal of material from the top surface down to a specified depth. Few studies exist on the engraving of metals, and there are even fewer studies concerning wood engraving. An important review was performed on laser engraving for various materials, including wood, by Patel *et al*. (2015), who investigated the influence of the process parameters (laser power, scanning speed, and laser frequency) on the engraving depth and surface roughness. Patel *et al*. (2015) acknowledged the work of Leone *et al*. (2009), who investigated wood engraving using a Q-switched diode pumped frequency doubled Nd:YAG green laser working at a wavelength of 532nm. The examined parameters were the pulse frequency, the beam speed, the number of laser scans, and the engraved depth. Experimental results showed that this type of laser can be successfully used to machine different types of wood, obtaining decorative drawing and 3D engraved geometries without burning. However, the authors stated that more studies are needed to correlate the wood species with appropriate process parameters, with a goal of achieving deep engraving without carbonization and still retaining a homogeneous carving.

Lin *et al*. (2008) investigated the effect of feed speed ratio and laser power on the engraved depth and colour difference of Moso bamboo lamina. It was found that the laser engraved depth became deeper for either a higher laser power or a lower feed speed ratio. Colour difference values increased at a lower feed speed ratio and a higher power and resulted in a brownish color in the engraved zone. The advantage of this study was that the engraved depth and colour difference values of Moso bamboo could be predicted and estimated by regression analyses.

Patel and Patel (2014) used surface roughness as the response for various parameters in laser engraving of stainless steel. The measured parameter was *Ra*. It was found that surface roughness increases with higher laser frequency and a lower engraving speed. Similarly, Pritam (2016) found that the surface roughness described by *Ra* and the engraving depth of stainless steel decrease with an increase in the scanning speed and a decrease in the laser power. The target was for a minimization

of surface roughness, while maximizing the engraving depth. To achieve deeper cavities, but with less roughness, the author recommends an increased number of laser scans at a lower power and a higher scanning speed.

In 1986, Barnekov *et al.* concluded that laser applications on wood have great potential, but they were not yet sufficiently explored. This statement is still valid today. One of the main factors influencing laser-wood interaction is the character of the wood itself, primarily its density, moisture content, extractives, and optical properties.

In summary, the effect of a laser beam on wood surfaces has scarcely been studied and no research has explored the effect of a laser beam on the surface roughness of wood.

OBJECTIVE

This paper aims to find the influence of varying some parameters of a CO_2 laser beam (laser power output and scanning speed) on the surface roughness of beech wood (*Fagus sylvatica*) for aesthetic applications such as decorative drawing. It is important to develop such information to understand the effect of the two parameters on the surface quality of beech when estimating superficial decoration with minimum engraving (below 1mm) using lasers.

MATERIAL, METHOD, EQUIPMENT

For superficial decorative engraving/drawing, a CO_2 laser (model SLG-4030 lsct with LaserCut software version 4.03 included, imported from China by SpotLine, Bucharest, Romania), with a wavelength of 10.6μm, a lens of 73mm focal length, and maximum output power of 40W was used. The scanning gap was 0.0254mm and the pulse frequency was 20000Hz.

For laser treatment, two parameters were varied, namely, the laser output power and the laser speed. Laser output powers used for this study were fractions from the maximum output power of 40W: 14% (5.6W), 15% (6W), 16% (6.4W), and 17% (6.8W). For simplicity, they are symbolized in this paper as L14, L15, L16, and L17, respectively. The tested scanning speeds were 100, 200, 300, 400, and 500mm/s. The target was to analyse their influence on the surface roughness occurring on laser scanned beech wood.

Four beech (*Fagus sylvatica*) specimens, conditioned at 20°C and 65% relative humidity of the ambient air, were prepared by first planing, then dimension cutting, then calibration with P60, and finally manual sanding with P100 grit size to their final dimensions of 340mmx100mmx8mm. The surfaces were semi-radial, which were preferred to tangential because they have less anatomical wood variation along the surface. The beech species was selected because of its availability.

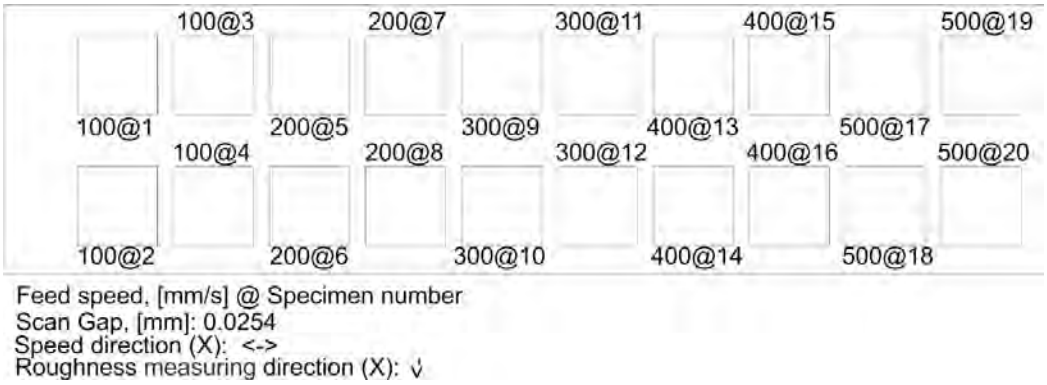

Fig. 1.
Schematic representation of a wood sample scanned with a laser beam on 25mmx25mm areas for five scanning speeds and four replicates for each speed.

Each of the four specimens was scanned with a different laser power output (from L14 to L17) as described above. On each specimen and for each output power, 20 areas (25mmx25mm) were laser scanned with scanning speeds from 100 to 500mm/s, so that for each scanning speed there were four replicates (Fig. 1).

Surface quality measurements were performed using a MarSurf XT20 instrument manufactured by MAHR Gottingen GMBH (Göttingen, Germany), fitted with a MFW 250 scanning head with a tracing arm in the range of ±500μm and a stylus with a 2μm tip radius and 90° tip angle, which measured the beech specimens, across the grain, at a speed of 0.5 mm/s, a low scanning force of 0.7mN, and a lateral resolution of 5μm.

From each laser processed area, one profile, 20mm long (Fig. 2a), was stylus scanned across the grain for surface roughness analysis of the combined effect of laser power and scanning speed, so that, for each laser power and scanning speed, four profiles were analysed. For all laser powers (L14 to L17) and five scanning speeds, there were 80 scanned profiles in total.

Those profiles and their roughness parameters were compared with similar 20-mm-long profiles of untreated wood, stylus scanned in the immediate vicinity of the laser modified areas (Fig. 2b), so that each laser power/scanning speed combination corresponded to one roughness profile from unmodified wood. This meant that for each specimen, five wood profiles were analysed, resulting in a total of 20 profiles for all laser powers examined (L14 to L17). Those profiles were used as references to observe any increase in surface roughness of laser scanned surfaces caused by the laser action.

To visualize those roughness differences, another group of profiles, 40mm long, were scanned so that they covered half a laser engraved region and half unprocessed wood (Fig. 2c). This meant five mixed profiles for each specimen, 20 mixed profiles in total.

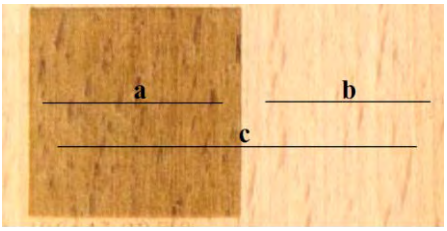

Fig. 2.
Details regarding the profiles measurement: a: profile from laser modified area (20mm long); b: profile from unprocessed wood (20mm long); c: mixed profile measuring laser scanned and unprocessed wood (40mm long).

Furthermore, the scanned profiles were processed with MARWIN XR20 software provided by the instrument supplier (Göttingen, Germany). The profiles were also saved as ASCII files with the possibility to be separately examined and visualized with MathCAD 2000 Professional (PTC, Needham, MA).

The procedure with profiles is standard. Any measured profile contains not only roughness, but also form errors and waviness (ASME B46.1 2009). A flat surface processed by sanding and then scanned by the laser is best characterized by surface roughness. This means that it is necessary to extract the roughness from the measured profile. Therefore, the form errors characterizing the machining accuracy were removed following the procedure given in ISO 3274 (1996). Furthermore, to obtain the roughness profiles, a filter should be applied to straighten the profile. However, it was found that common filters, such as the simple Gaussian filter, can alter the measured profile, causing some artificial "push-up" in areas with deep wood pores. This drawback can be avoided by using a more recent and robust filter called RGRF (robust Gaussian regression filter) contained in ISO 16610-31 (2010). This filter was tested and found useful for wood surfaces because it is more robust than the simple Gaussian filter and does not introduce bias related to wood anatomy (Gurau *et al*. 2006).

This filtering procedure is quite new for wood surfaces and has the advantage of providing a more reliable result regarding the surface roughness of wood. The cut-off used in this research was 2.5mm, as recommended in previous studies by Gurau *et al*. (2006).

After the roughness profiles were correctly obtained, a range of roughness parameters was calculated for profiles, such as *Ra*, *Rq*, and *Rt* from ISO 4287 (1997) and *Rk*, *Rpk*, and *Rvk* from ISO 13565-2 (1996). Their mean values and standard deviations were also calculated.

Mean parameters *Ra* (the arithmetical mean deviation of the assessed profile) and *Rq* (the root mean square deviation of the profile) are common roughness indicators, but they alone do not provide sufficient information about wood surface topography. Furthermore, it is expected that they will be influenced by deep wood anatomical irregularities. Similarly, *Rt* (the total height of the profile) is expected to be sensitive not only to processing with the laser but also to variations in local wood anatomy.

Rk (the core roughness depth), *Rpk* (the reduced peak height), and *Rvk* (the reduced valley depth) are interesting parameters. They are calculated by following a standard procedure in ISO 13565-2 (1996), where the surface irregularities are first ranked in descending order. Then, the region with the highest concentration of data points is delimited by *Rk*, which is also the parameter that is

least influenced by wood anatomical irregularities and whose values should approximate the contribution of processing (the marks caused by the processing tool). In the case of this study, the processing that preceded the laser scanning was sanding with grit P100. It is assumed that the grit particles have left specific sanding traces on the samples overlapping on the wood anatomical irregularities. Then, the laser affected the sanded beech surface by increasing the magnitude of peaks and valleys in the initial surface and presumably increasing the core roughness *Rk*. However, no study has explored how much laser power combined with a variation in the scanning speed contributes to this topographic change in the wood surface.

Rpk is a parameter that gives a measure of the magnitude of raised fibres above the core roughness, while *Rvk* measures the magnitude of deep irregularities extending below the core roughness (in the case of wood, associated with anatomical valleys). It is expected that the laser will influence all these parameters: *Rk*, *Rpk*, and *Rvk*. Therefore, a composed parameter comprising the three regions was also used in this research: *Rk* + *Rpk* + *Rvk*.

In addition to calculating the roughness parameters, the measured data were imported into MathCad, and roughness profiles were visualized to compare the magnitude of irregularities from unprocessed beech with those where the laser was used.

The effect of the laser power output on the core surface roughness was made visible by calculating the location of thresholds that delimit the core roughness from the isolated peaks and valleys with a method derived from ISO 13565-2 (1996) and described in detail by Gurau ***et al***. (2005).

RESULTS AND DISCUSSION

Laser scanned images for various laser power-scanning speed combinations are contained in Table 1.

Table 1

Example of laser scanned areas for various laser power-scanning speed combinations

	100 mm/s	200 mm/s	300 mm/s	400 mm/s	500 mm/s
L17					
L16					
L15					
L14					

Roughness parameters mean values for surfaces scanned with laser powers L14 to L17 combined with scanning speeds from 100 to 500mm as well as those measured from unprocessed beech surfaces are contained in Table 2.

Table 2

Mean roughness parameters for beech scanned with various laser power outputs and scanning speeds in comparison with unprocessed surfaces. In parenthesis, standard deviation values

Processing	**Laser scanning speed (mm/s)**	***Ra* (µm)**	***Rq* (µm)**	***Rt* (µm)**	***Rk* (µm)**	***Rpk* (µm)**	***Rvk* (µm)**	***Rk+Rpk+Rvk* (µm)**
L14	100	11.8 (0.67)	15.8 (0.78)	112.3 (9.64)	33.6 (2.08)	10.2 (0.96)	24.2 (1.04)	68.0 (2.07)
	200	10.0 (0.32)	13.1 (0.44)	91.8 (5.81)	29.9 (1.56)	8.6 (0.75)	19.1 (1.20)	57.5 (2.51)
	300	9.9 (0.31)	12.9 (0.54)	83.6 (5.57)	29.1 (2.22)	7.8 (1.57)	19.5 (2.68)	56.5 (1.70)
	400	10.4 (0.74)	13.7 (0.95)	89.6 (7.17)	31.2 (2.90)	7.6 (1.75)	21.5 (2.45)	60.3 (4.53)
	500	9.6 (0.34)	12.3 (0.62)	80.9 (12.16)	29.9 (1.41)	10.4 (1.00)	15.7 (2.26)	56.1 (1.84)
L15	100	12.9 (0.91)	17.2 (1.49)	131.0 (20.70)	38.8 (2.94)	10.0 (0.90)	26.8 (4.64)	75.6 (6.90)
	200	10.9 (1.12)	14.6 (1.55)	111.0 (18.71)	30.7 (2.82)	10.1 (2.73)	22.6 (1.39)	63.4 (6.17)
	300	10.4 (1.08)	13.4 (1.58)	89.8 (18.03)	32.9 (3.60)	8.8 (2.35)	17.9 (2.79)	59.5 (6.77)
	400	10.0 (1.03)	13.0 (1.73)	86.3 (12.49)	30.7 (2.77)	7.5 (0.64)	18.7 (4.83)	56.9 (7.13)
	500	10.5 (0.48)	13.6 (0.69)	93.1 (16.84)	32.3 (3.05)	8.2 (1.63)	19.0 (3.27)	59.5 (5.07)
L16	100	16.1 (1.46)	20.9 (1.90)	130.3 (16.45)	48.2 (6.05)	12.7 (1.52)	29.4 (4.09)	90.4 (10.86)
	200	11.3 (0.84)	14.8 (0.97)	105.7 (9.14)	33.1 (4.06)	8.1 (1.25)	21.8 (3.35)	63.0 (4.09)
	300	11.0 (0.59)	14.2 (1.05)	94.4 (5.23)	34.0 (1.55)	8.9 (1.94)	18.9 (2.84)	61.7 (3.73)
	400	10.4 (0.79)	13.7 (1.31)	95.2 (2.24)	31.1 (1.57)	9.5 (1.85)	19.6 (3.91)	60.2 (5.60)
	500	10.4 (0.71)	13.7 (1.20)	91.9 (16.97)	31.3 (2.01)	11.2 (2.39)	18.9 (4.37)	61.4 (5.50)
L17	100	22.0 (2.91)	27.8 (3.62)	170.7 (22.36)	69.5 (10.91)	18.2 (2.74)	33.8 (4.95)	121.4 (17.43)
	200	13.5 (0.29)	17.3 (0.38)	115.4 (4.59)	41.6 (1.31)	12.4 (1.64)	23.3 (0.68)	77.2 (1.94)
	300	10.8 (0.69)	13.9 (0.99)	96.8 (16.98)	33.4 (1.77)	11.5 (3.15)	17.6 (1.74)	62.5 (4.94)
	400	11.0 (0.81)	14.0 (0.95)	87.9 (3.25)	34.8 (3.15)	9.7 (1.12)	18.8 (2.05)	63.3 (3.29)
	500	10.7 (0.51)	13.9 (0.59)	103.9 (17.93)	33.2 (3.15)	11.9 (1.28)	19.2 (0.65)	64.2 (3.24)
Beech unprocessed	Mean from all areas (20 values)	**10.2 (0.84)**	**13.3 (1.16)**	**86.7 (7.81)**	**30.9 (2.80)**	**7.7 (1.26)**	**18.9 (2.92)**	**57.5 (4.66)**

Among the roughness parameters, the best correlation with the laser power and scanning speed was obtained for the depth of the profile *Rk* + *Rpk* + *Rvk*.

Fig. 3 shows the variation of the combined roughness parameter *Rk* + *Rpk* + *Rvk* with the laser power and scanning speed in comparison with the reference, for unprocessed beech. The best correlations were obtained for a third-order polynomial, which was fit for all laser power data points. The coefficients of correlations R^2 were high for all curves, with the highest values recorded for the laser power L17.

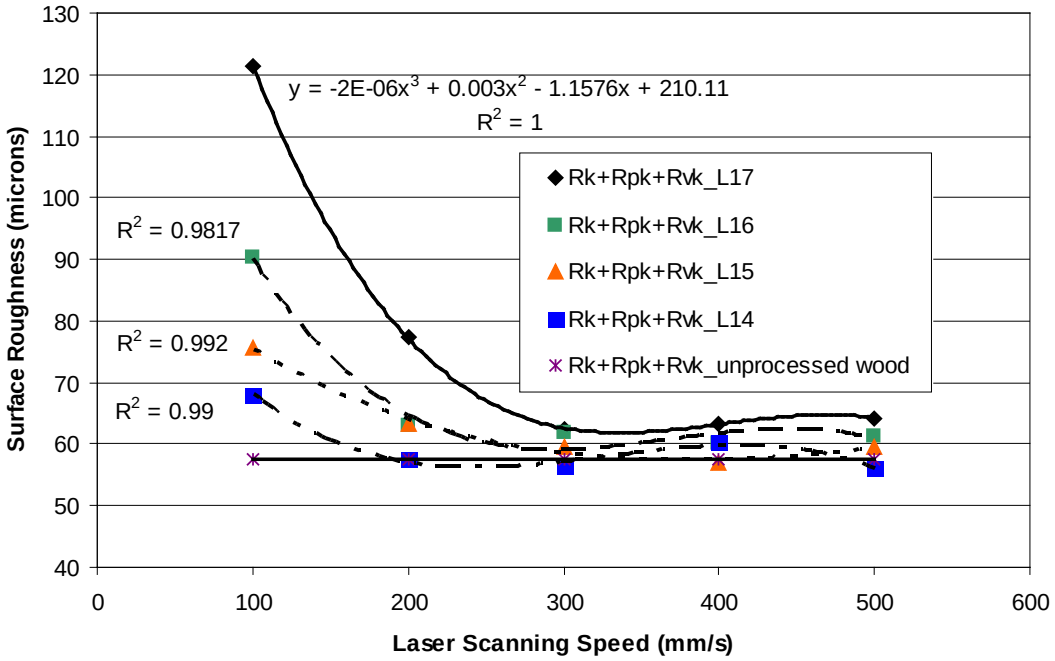

Fig. 3.
Variation of the depth of the profile (Rk + Rpk + Rvk), with the laser power and laser scanning speed in comparison with unprocessed beech (solid horizontal line).

From Table 2 and Fig. 3 , it can be seen that the roughness values increased with the laser power.

The laser scanning speed had a strong influence on the surface roughness values. The highest values were recorded for all laser powers at a scanning speed of 100mm/s, which in case of high powers, L16 and L17, caused burning of the surface, which was perceived as a strong dark colour and as a level difference from the surrounding wood (engraving effect less than 1mm).

It was interesting to evaluate how much roughness change was caused by the laser action on wood. In Table 3, the extreme cases of laser processing effect on wood were included. The minimum effect was obtained for laser power L14 combined with a scanning speed of 500 mm/s, which was compared with the roughness of wood measured in the proximate vicinity (as shown in Fig. 2 a and b). The increase in roughness in the laser processed area is presented as a percentage. Similarly, the maximum effect on wood was produced by the combination of laser power L17 and a scanning speed of 100mm/s.

Table 3
Minimum and maximum effect of laser power-scanning speed on surface roughness of wood in comparison with neighboring unprocessed wood

	Laser power L14 at 500 mm/s	Neighbouring wood	Roughness increase (%)	Laser power L17 at 100 mm/s	Neighbouring wood	Roughness increase (%)
Ra (µm)	9.6	9.59	0.10	22	9.44	133.05
Rq (µm)	12.3	12.18	0.99	27.8	12.1	129.75
Rk (µm)	29.9	29.6	1.01	69.5	29.3	137.20
Rpk(µm)	10.4	7.91	31.48	18.2	7.04	158.52
Rvk(µm)	15.7	15.4	1.95	33.8	17.02	98.59
Rk + Rpk + Rvk (µm)	56.1	52.91	6.03	121.4	53.36	127.51

From Table 3, the greatest effect of laser action on wood was observed on *Rpk* (surface fuzziness), which increased by 31.48% for laser power L14 and by 158.52% for laser power L17. An increase in roughness was observed for all roughness parameters, but the parameter *Rk + Rpk + Rvk* showed a strong cumulative effect: surface roughness of beech wood laser scanned with L17 at a scanning speed of 100mm/s increased the surface roughness by 127.51%, corresponding to an absolute height difference of approximately 68 μm. The value *Rk* increased by 137.20%, corresponding to an absolute difference of 40 μm. For finishing applications, this surface is considered very rough. The rougher the surface is, the more finish (lacquer) it will absorb. It must be noted that the measurement was performed inside the laser engraved area and the parameters did not measure the level difference from the surrounding wood (the depth of the engraved area).

In the case of the laser power L14, with scanning speeds of 200 to 500 mm/s, the effect on surface roughness was obscured by local wood roughness (wood anatomical irregularities and irregularities from previous processing by sanding) (Table 2 and Fig. 3). In this domain of scanning speed, the roughness parameters for L14 had a pendular trend with respect to the wood roughness, possibly because of local wood anatomical variation.

For L15, the roughness decrease was sharp for scanning speeds of 100 and 200mm/s, but with a tendency to stabilize from a scanning speed of 300mm/s and, as above, with roughness parameters obscured by wood roughness from scanning speeds from 300 to 500mm/s.

For laser powers L16 as well as for L17, the decrease in roughness for all parameters was sharp, from a scanning speed of 100 to 300mm/s, then the values were rather constant as the scanning speed increased but always with higher roughness values than for unprocessed wood (Table 2 and Fig. 3).

Compared with the laser power L14, for a scanning speed of 100mm/s, the core roughness *Rk* for L17 doubled, while *Rk* increased by 43.5% for L16 and by 15.7% for L15. This shows that the laser power has a strong impact on surface roughness for low scanning speeds.

The strong influence of laser scanning speed on the surface roughness of beech wood can be seen for laser power L17 in Fig. 4, where a mixed profile is presented with approximately half the length from the laser scanned surface and half from unprocessed wood. The first half of the profile shows a high magnitude of irregularities in comparison with the second half, where wood was left unprocessed. Those profile regions were separated and presented in Fig. 5 and Fig. 6, respectively, both containing thresholds which delimit the core roughness. It can be observed that the core roughness of beech processed with L17 was more than double the core roughness of unprocessed beech (see also *Rk* in Table 3).

In contrast, the core roughness of a profile taken from beech processed with laser power L14 at a scanning speed of 100mm/s in Fig. 7 shows only slightly higher values as compared with unprocessed wood core roughness (Fig. 6). The value *Rk* for laser power L14 and a scanning speed of 100mm/s was 8.6% higher than *Rk* for unprocessed beech (Table 2).

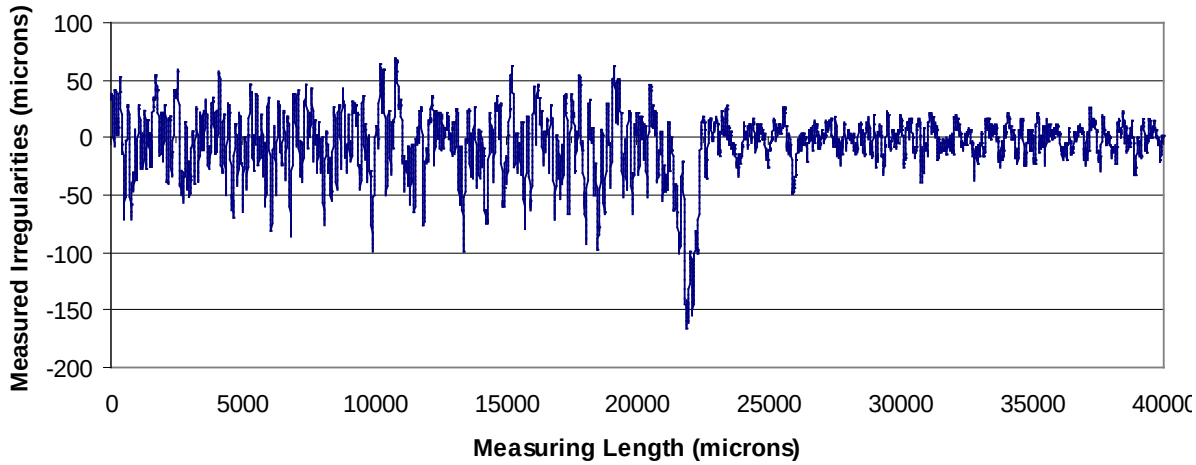

Fig. 4.

Mixed profile measuring approximately half length from beech engraved with L17 laser power at 100mm/s scanning speed and half from unprocessed beech. Profile length: 40mm.

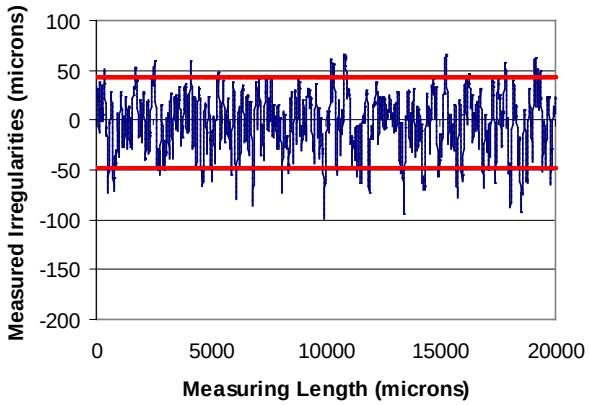

Fig. 5.
Detail of the mixed profile from Fig. 4., representing only the laser scanned region (left part) of the profile (laser power L17 and 100mm/s scanning speed). The red lines represent thresholds delimiting the core roughness. Profile length: 20mm.

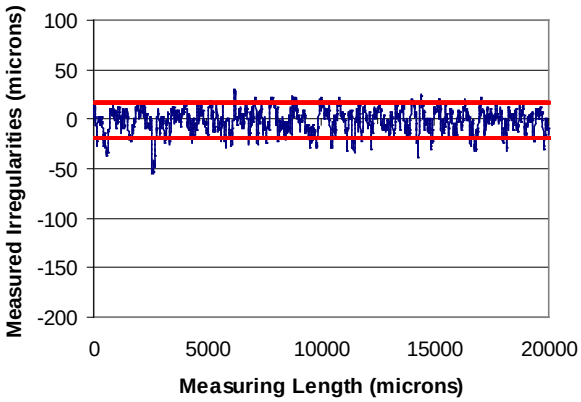

Fig. 6.
Detail of the mixed profile from Fig. 4., representing only the unprocessed wood region (right part) of the profile. The red lines represent thresholds delimiting the core roughness. Profile length: 20mm.

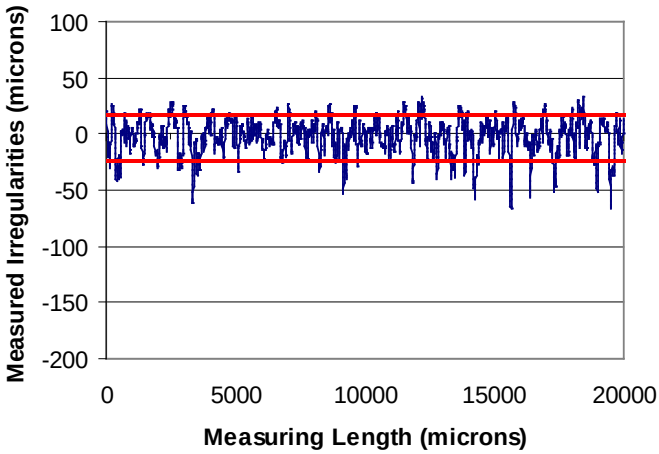

Fig. 7.
Profile measured from beech scanned with L14 laser power at 100mm/s scanning speed. The red lines represent thresholds delimiting the core roughness. Profile length: 20mm.

Perhaps, when choosing an optimum laser power in conjunction with a scanning speed, one should consider the occurrence of a minimum surface roughness to which an important colour change corresponds.

L16 and L17 laser powers caused wood burning for lower scanning speeds of 100 and 200mm/s. For each of the laser powers, for speeds of 300 to 500mm/s, the surface roughness was similar, while the colour difference slightly decreased.

This study can be extended for higher laser powers and different combinations of scanning speeds to understand how these parameters modify the surface roughness.

CONCLUSIONS

The surface roughness increased with the laser power and decreased with the scanning speed. The highest changes as compared to unprocessed wood were obtained for the lower speeds of 100 and 200mm/s. For speeds higher than 300mm/s, the surface roughness was nearly the same.

The laser powers L16 (6.4 W) and L17 (6.8 W) caused surface burns and an engraving effect (depths below 1mm).

The best descriptor of the surface roughness change due to laser action was the composed parameter *Rk* + *Rpk* + *Rvk*. The most pronounced topographic effect of the laser on wood was an

increase in surface fuzziness as measured by *Rpk*, combined with an increased core roughness (*Rk*) and deeper *Rvk*.

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SECTION 4. WOOD-BASED MATERIALS

CT SCANNING OF CAPILLARY PHENOMENA IN BIO-BASED MATERIALS

José COUCEIRO
Luleå University of Technology (LTU)
Wood Science and Engineering
SE-93187 Skellefteå, Sweden
E-mail: jose.couceiro@ltu.se

Margot SEHLSTEDT-PERSSON
Luleå University of Technology
Wood Science and Engineering
SE-93187 Skellefteå, Sweden
E-mail: margot.sehlstedt-persson@ltu.se

Lars HANSSON
Norwegian University of Science and Technology (NTNU)
Department of ICT and Natural Sciences
N-1517, 6025 Ålesund, Norway
E-mail: laha@ntnu.no

Olle HAGMAN
Luleå University of Technology
Wood Science and Engineering
SE-93187 Skellefteå, Sweden
E-mail: olle.hagman@ltu.se

Dick SANDBERG
Luleå University of Technology
Wood Science and Engineering
SE-93187 Skellefteå, Sweden
E-mail: dick.sandberg@ltu.se

Abstract

X-ray computed tomography (CT) is a powerful tool for the non-destructive study of dynamic moisture processes in wood and other bio-based materials. In the CT facilities at Luleå University of Technology, it is possible to study wood-moisture relations such as water absorption, drying and related material deformation under a temperature- and humidity-controlled environment.

An increase in the use of bio-based materials in building construction has led to an increased interest in capillary phenomena in these materials, because of an increasing number of moisture-related damage in timber and hybrid-timber buildings. This article shows some examples of how different bio-materials used in construction interact with liquid water over time. The overall purpose has been to develop the CT technique as a powerful tool for the determination and visualization of capillary flow that can be a base for modelling and an increased understanding of moisture flow in new bio-based building materials.

Early-stage observation of the behaviour of different traditional and new bio-based building materials shows that CT scanning, combined with image processing, has a high potential to be used in performing non-destructive and non-contact tests that can help to increase the knowledge of water-material interactions and develop building materials with an optimized performance.

Key words: *X-ray computed tomography; wood; water flow.*

INTRODUCTION

The use in construction of building materials based on residues from forest and agricultural activities is growing as society moves towards a bio-based economy. This represents an opportunity to turn waste material from these activities into profitable by-products. One common use for this type of material is as thermal insulation for buildings. The durability and functionality of bio-based insulation materials depends to a high degree on their behaviour and interaction with water. Capillary water is of crucial importance for the durability and long-term performance, because of the severe damage that

can occur in a relatively short time. Current knowledge about material-water relations for many bio-based materials is inadequate, especially for the newest products such as insulation materials from recycled paper, flax, jute and wood fibres. Furthermore, the standards that are accepted for the determination of certain physical properties may not be easily applicable to newly developed materials with a composition and structure that differ substantially from the products that they may substitute and for which the standards are developed. For that reason, new ways of studying the interaction of new bio-based materials with moisture is of crucial importance for their durability and performance as competitive building materials.

In their natural state, bio-based materials from the forest and the agricultural sector are biodegradable and strongly hygroscopic, in contrast to materials from non-renewable sources. When new bio-based materials are introduced into e.g. the building sector, questions related to how the materials and structures interact with moisture will arise.

A better understanding of material properties is therefore necessary for architects, designers and other professionals involved at an early stage in the design and development of buildings. An example of poor performance was the external wall insulation system EWIS in Swedish single-family houses that quickly led to severe moisture and mould problems, and also to health problems for people living in the houses (Swedish High Court Judgement 2015). Experience from history of how wood in particular has been used in an unfortunate way is frightening, both in construction and in the way it has been handled during the building phase. The most common problems involve moisture-related damage, and such problems are often presented as a serious objection to an increase in the use of bio-based materials and they show the need for further development.

Another crucial aspect is that the climate change is expected to lead to new material requirements particularly with regard to their functioning under an increased moisture stress during extreme rainfall and floods.

Capillary water transport in bio-based materials is a particularly critical feature that can lead to unfavourable moisture levels. Unacceptable discoloration caused by mould growth and fungal decay, critical delamination of coating or adhesive, dimensional instability, deterioration in mechanical performance and in insulating capacity are examples of building material properties flawed by poor moisture control (Dvinskikh *et al.* 2011).

Capillary flow is three-dimensional and non-gradient driven. Experimental studies and models for capillarity are not trivial, since capillarity is governed by small-scale variations on a microstructural scale in the material. Species-related properties also have a significant influence on the capillary flow. Monocotyledons, for example, have a special layer on their external surface consisting of waxes that dramatically influence the contact angle and thus the capillary properties (Barthlott *et al.* 1997). This is of great importance for the usability of Reed canary grass and other agricultural raw materials in construction materials (Trischler and Sandberg 2014).

Capillary-driven flow in a hygroscopic material has traditionally been modelled as a diffusion process. It is well known, nevertheless, that it is not actually a proper diffusion process. An increasing number of experimental results indicate that the diffusion approach cannot correctly describe some essential parts of the process (see e.g. Salin 2006), and the knowledge level of the mechanism of capillarity in many bio-based materials is low. One approach for modelling capillary flow is the percolation approach where the material is viewed as a porous material with cavities of different sizes (Stauffer and Aharony 1994). The influence of time and how time enters into capillary flow are, however, somewhat unclear, since flow cannot be instantaneous. Capillary flow has been studied relatively thoroughly for wood (Petty 1974, Spolek *et al.* 1981, Perre and Turner 2001, Segerholm and Claesson 2008, Zelinka *et al.* 2016). Several other hygroscopic materials have been studied by e.g. Peishi and Pei (1989), Segura and Toledo (2005), and Zhu *et al.* (2012). Nearly all these authors indicate that a basic understanding of the underlying mechanisms for capillary flow in the bio-based materials studied is deficient, and this leads to a poor output from modelling work on capillary flow. This is an argument for the more detailed study of the capillary flow phenomenon presented in this paper.

For a bio-based material to be competitive in most building applications, it is necessary to find means to enhance its performance and service life. A possible way to accomplish this is through modification. Three fundamentally different wood modification methods have recently been commercialized: acetylation, furfurylation and thermal modification (Hill 2007, Navi & Sandberg 2012).

At the Wood Science and Engineering department at LTU in Skellefteå a score of PhD theses have been produced based on research that has been built up around the use of computer tomography (CT) in wood. The results of these CT studies have totally changed the understanding of water flow in the capillary regime during wood drying (Morén and Sehlstedt-Persson 2000, Wiberg 2001, Sehlstedt-Persson *et al.* 2006, Schepers *et al.* 2007, Johansson and Kifetew 2010, Hansson

and Cherepanova 2012, Vikberg *et al* 2012). Nevertheless, there is a lack of knowledge regarding bio-based materials, which are particularly complex due to the fact that they are both porous, and also consist of fibre walls that are hygroscopic and absorb water vapour from the air. For the same reason, the loss of capillary water from bio-based material during drying must be explored in detail. In addition to this lack of knowledge in the field, this project deals with the lack of standardised tests to study capillary absorption phenomena in porous materials. Thus, the first efforts are being focused on the design of the experiments.

The long-term vision driving this project is that by-products from the forest or agricultural biomass will be used to replace materials in building applications where fossil and non-renewable materials are currently being used, and that the use of bio-based materials will increase to a level which supports a sustainable development.

Computed tomography

Since it was introduced in the 1970s, X-ray computed tomography (CT) has proven to be a powerful tool in the medical field and its use in materials science is now widespread. It is an imaging technique based on measurements of the amount of X-ray radiation that is able to pass through a body of a given material, a property that is defined by the attenuation coefficient of the material (Kalender 2011). The theoretical background of CT lies in Lambert-Beer's law, which shows an exponential relationship between the intensity of the radiation and the attenuation coefficient:

$$I = I_0 e^{-\mu d} \quad (1)$$

where: I is the intensity of the transmitted X-ray beam, I_0 is the intensity of the incident X-ray beam, μ is the linear attenuation coefficient of the material along the transmission path and d is the thickness of the body.

CT images are in a grey scale and, for most biological materials, the grey scale values are almost linearly related to density, being darker for lower density and brighter for higher density

OBJECTIVES

The purpose of this paper is to show ways in which different bio-materials used in construction interact with liquid water over time. The main parameter taken into account is the absorption of liquid water above the water level when the material is partially submerged. Other parameters like bound water gradient, material structure and a comparison between modified and unmodified materials are also considered and studied.

MATERIALS, METHODS AND EQUIPMENT

The studied materials were classified in two groups: (1) "solid materials", i.e. wood and traditional wood-based materials. Even though these materials are actually porous, they are referred to here as solid materials to distinguish them from the materials in the other group. (2) Porous materials, such as various low-density insulation materials produced from forest and agricultural by-products, recycled newspapers, pulp, jute, wood fibres, etc. As an example of modified wood, specimens of thermally modified timber have been included in the tests. The specimens were obtained as boards (15 to 30mm thick) or panels (45 to 65mm thick) and cut to a dimensions of 230x160mm. The specimens were conditioned at a temperature of 20°C and 60% RH for seven days before the tests started.

A medical CT-scanner Siemens Somatom Emotion Duo with a field of view of 500x500mm^2 represented in a 512x512 pixel image, which gives a resolution of 0.98mm, was used. By applying different reconstruction algorithms, images with a higher resolution can be generated, up to 0.1mm, but with the side-effect that properties of the images such as noise and sharpness are altered. The scanning depth ranges from 1mm to 10mm, thus the smallest voxel that can be represented in an image is 0.1x0.1x1mm^3. This piece of equipment was the main tool around which the research took place. The scanning time of this scanner is around 1s for a single scan. In this case, the scanning is performed in a spiral along a distance of about 900mm, which produces nearly 300 single 3mm thick scans that are taken in a continuous manner. This process takes about 70s. In order to perform experiments in a controlled and monitored environment, a drying chamber that works in combination with the scanner was used, allowing the scanning of the interior of the chamber as the temperature and relative humidity were controlled and regulated with time.

A rig of acrylic plastic in which specimens were held in a vertical position was manufactured specifically for these experiments (Fig. 1). The rig allows water to be filled to a certain level so that the specimens were partially submerged in a constant level of water (around 15mm) and so that they

could be scanned. Three plastic balls filled with water were placed in the rig (visible in Figs. 1 and 2), for spatial coordinate references in the evaluation of the CT images. CT scans were performed periodically in order to observe the transport of water within the samples. These images were processed so that both visualization tools and models of the phenomena can be developed at a further stage in the project.

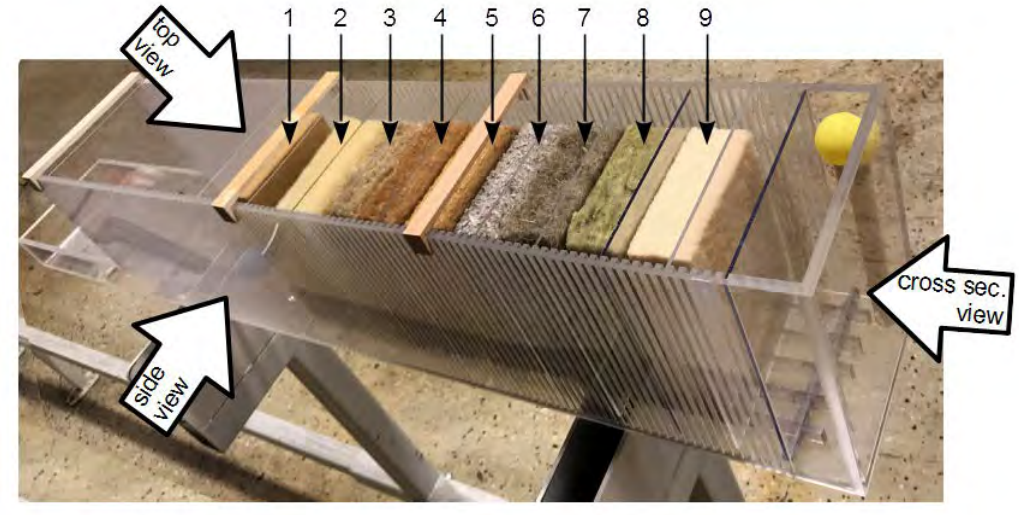

Fig. 1.
Experimental setup. Tested bio-based insulation material in the rig are: (1) Pavatherm combi (wood fibers), (2) Glass fiber, (3) Thermohanf Premium (hemp), (4) Thermojute (jute), (5) STEICOflex (pine wood fibers), (6) iCell (newspaper), (7) Isolina (linen), (8) Stone wool, (9) Thermocell (pulp).

RESULTS AND DISCUSSION

Fig. 2 shows how different materials can be visualized simultaneously and how the water uptake can be compared. The image shows clearly the different levels of water absorption in three different materials (Fig. 2b,c,d), and the rise of capillary water to a higher level inside fibres (Fig. 2d). In this kind of material made of vegetal fibres and particles, a large moisture gradient can be seen rising (Fig. 2b,c). This could be due to capillary absorption in fibres with a small diameter that are not visible at this resolution level, which would allow a higher level of capillary absorption, or it could be due to a rise in the moisture content level in the cell-wall material that remains.

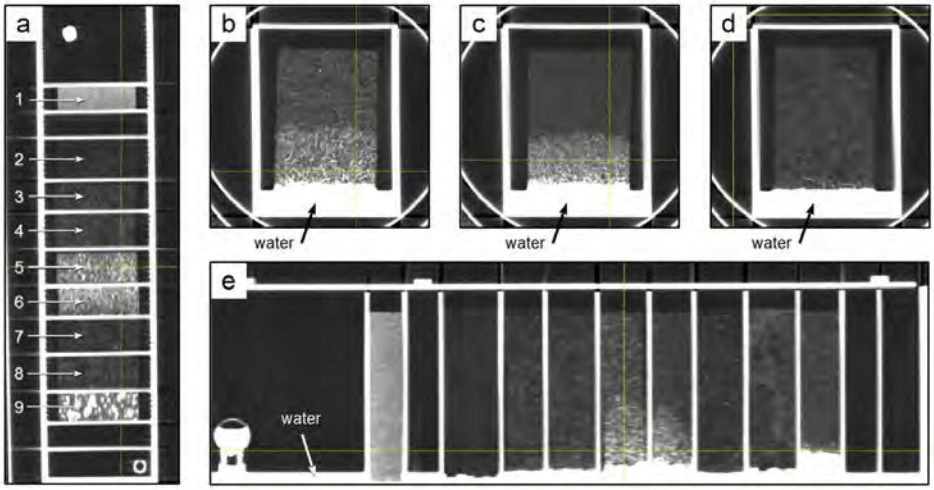

Fig. 2.
CT images of the experimental setup: (a) top view of the rig including 9 different insulating materials according to Fig. 1, (b-d) Cross section view of three different insulation materials submerged in water for 24h: (b) pine wood-fibre (Fig. 1 (5)), (c) recycled newspaper (Fig. 1 (6)), and (d) jute panel (Fig. 1 (4)). (e) side view of the rig. Cross section, top and side views are according to Fig. 1.

In the group of solid materials, various interesting and unexpected phenomena were seen. Fig. 3 shows the difference in water uptake (white colour) between aspen heartwood and sapwood within a single piece, where it can be seen that sapwood absorbs more water than heartwood.

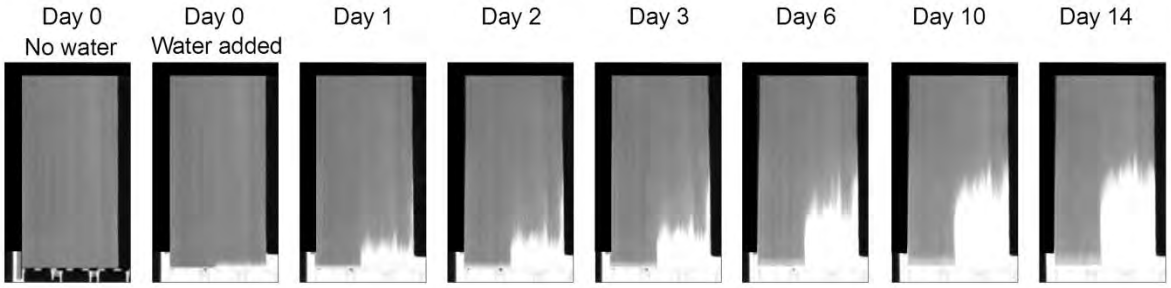

Fig. 3.
Water absorption (white area) in an aspen specimen containing sapwood and heartwood in cross section view according to Fig. 1.

Thermal modification is a process where wood is heated to about 200°C in order to decrease the hygroscopicity and increase the dimension stability of the material. Water absorption in solid aspen and birch wood gets dramatically reduced after thermal treatment. This is clearly visible in the CT images and it is possible to follow as a function of time (Fig. 5).

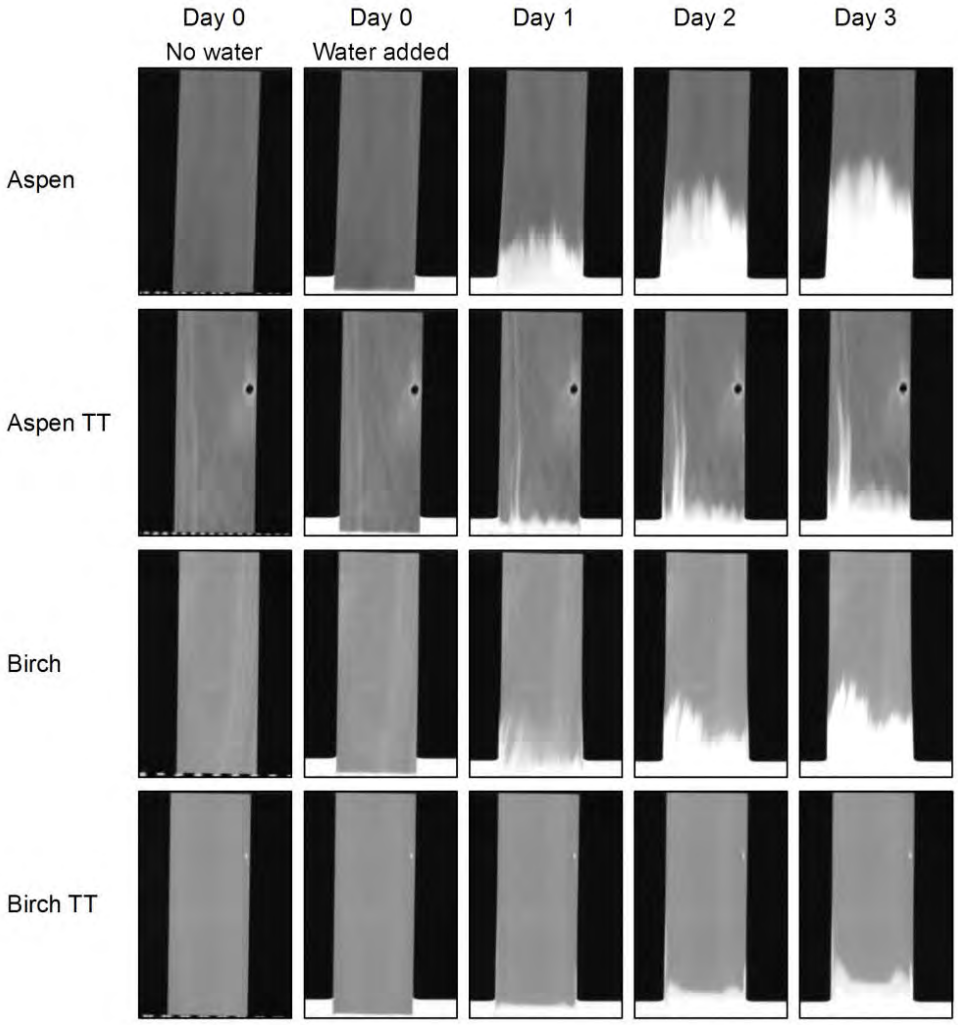

Fig. 5.
Water absorption (white area) in unmodified and thermally treated (TT) aspen and birch in cross section view according to Fig. 1.

Future work

More tests are planned using specimens from both groups of materials and different parameters and experimental setups. The over-all aim of the project is to introduce CT scanning in temperature- and humidity-controlled environments, combined with image processing, as a tool for continuous non-destructive and non-contact 4D-studies of bio-based building materials exposed to water. The management and editing of the large amount of images that the experiments provide are a technical challenge for future experiments. Nevertheless, the work in programming and image analysis has already started so that it can be applied in the next stage of the project. The objectives of the future work are the visualization of dynamic flow in real-time (which involves developing image-processing algorithms), relating the dynamic flow process to the characteristics of the material under study and, finally, the installation of demonstration sites where bio-based materials can be exposed and monitored so that data from in-situ sensors can be validated against the results of the CT experiments.

CONCLUSIONS

X-ray computed tomography (CT) scanning combined with image processing is a powerful tool for continuous non-destructive and non-contact 4D-studies of water flow in bio-based building materials. It reveals capillary phenomena and provides data that can help an understanding of these phenomena. Within a single specimen of insulation material it is possible to appreciate differences in the behaviour of different parts in materials such as those formed by larger fibres, which clearly show how the capillary absorption is higher within these fibres than between them. In solid wood, the different levels of water flow between heartwood and sapwood can be studied. Knots, sapwood-heartwood, fibre orientation and other anatomical characteristics can also be studied in relation to their interaction with water. Water flow in thermally modified timber (birch and aspen) is less than that in unmodified timber, and the moisture gradient formed above the liquid water is also reduced. The examples given in this paper provide an overview of the potential of CT scanning for testing different materials with regard to their interaction with water, which in turn opens up new opportunities for improving the performance of newly developed building materials.

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COMPRESSION STRENGTH OF VENEER REINFORCED SANDWICH PANELS

Zsigmond VAS
University of Sopron, Simonyi Károly Faculty of Engineering, Wood Sciences and Applied Art
Bajcsy-Zs. street nr. 4, 9400 Sopron, Hungary
Tel: 0036 99 518 179, E-mail: vas.zsigmond@uni-sopron.hu

Levente DÉNES
University of Sopron, Simonyi Károly Faculty of Engineering, Wood Sciences and Applied Art
Bajcsy-Zs. street nr. 4, 9400 Sopron, Hungary
Tel: 0036 99 518 231, E-mail: denes.levente@uni-sopron.hu

Abstract

*Sandwich structures, originally developed and used in aerospace industry have found applications in other industry branches such as marine, automotive and sports goods industries. Typical sandwich structures are made of a low-density core material bonded with thin, strong skins at top and bottom resulting in very efficient load bearing structures. This paper describes the evaluation processes of physical and mechanical properties of a recently developed veneer based sandwich composite. In the honeycomb-like construction of the sandwich panels, the veneer reinforcements provide strength and stiffness, sets the final dimensions in thickness while the polyurethane foam has lateral supporting effect. The effect of honeycomb geometry and number of constituent layers on the compressions strength was analyzed by 3-level, 2-factor robust parametric design (**3^{II}** RPD). Standard test results confirmed the improved compression load resistance; while the cylindrical geometry over performed the sinusoidal shaped core reinforcements. The intended use of the developed composites include carrier substrates for counter tops, interior door leaves, indoor heat insulating and acoustic insulation panels, as well as structural insulated panels (SIP).*

Key words: *sandwich structures; veneer honeycomb; compression strength; corrugated core.*

INTRODUCTION

In recent years, product development trends are characterized by high degree of variability, individuality, the use of newly developed materials (composites, plastics, structured surfaces, functional materials, etc.), and combination of different material types. Due to this high variability during the structure design and dimensioning a careful attention should be paid for these heterogeneous products comprising two or more materials with different properties in order to fulfill the esthetical, strength, durability, stability and other requirements. Sandwich structures, as a response to the above-enumerated requests, were used in aerospace applications for the first time in World War II. The wings and the fuselage were constructed of plywood with balsa core. A sandwich structure is usually a 3-ply construction comprising simplex or complex alternating layers which are bonded to form a structural unit. The main advantage of sandwich structures over traditional ones is the high strength-to-weight ratio, good heat and acoustic insulation properties. The skin layers are made of high strength materials which are relatively thin, although they respond for the structure's overall load bearing capacity because of the high tension and compression power. The core layer is generally made of low strength and lightweight material. The core task is to separate and space the skins and to bear the shear forces according to Kovács (1975). In a sandwich structure generally the bending loads are carried by the force couple formed by the facesheets and the shear loads are carried by the lightweight core material (Nguyen et al. 2005).

Nowadays sandwich panels have many application fields like in constructions, metal, plastic and wood industry. Because of the high complexity of sandwich structures many research works have been done to determine and model their physical and mechanical properties. A study by Aktay (2007) showed that the compression strength of aluminum with Nomex honeycomb core structure depends on the cell size and wall thickness regardless of the material properties. Fiber-reinforced polymer honeycomb sandwich beams' behavior against torsional loads were studied by Davalos (2008) performing mechanical measurements and finite element modeling. Chen (2012) in his paper demonstrated the relationship between surface and core thickness ratio on sandwich panels with MDF surface layers and paper honeycomb core. According to results the lower the ratio the higher the modulus of elasticity and modulus of rigidity. The increase is significant when the thickness ratio is less than six. Multilayer sandwich panels using cork agglomerate as core material and Aleppo pine

wood veneer as face sheets were developed by (Lakreb et al. 2015). The cork agglomerate provided a high performance under perpendicular compression, while the wood layers protected the core material and increased its mechanical strength. Modelling results on the mechanical behavior of the sandwich structures have been reported by Borsellino et al. (2004) using commercial ANSYS code in order to model the sandwich structures in compressive, shear and flexural loadings. The static-mechanical behavior of the composite structure was well approximated by numerical simulations in the elastic zone but in the plastic regime, there was not a compatibility with the experimental data. The bending creep as a function of time was studied by Chen (2011). The results show the influence of core geometry and wall thickness as well as the thickness of surface layers and the material properties of the surface on creep. In an article by Wang (2009) sandwich panels with paper honeycomb and cardboard surface were used to study the energy absorption. Petras (1999) examined the failure mode of Nomex honeycomb beams at three point bending tests. Fatigue properties of sandwich beams with carbon woven/epoxy skins and Nomex hexagonal honeycomb core under 3-point bending cyclic loading were investigated by W. Boukharouba et al. (2014). The obtained data has shown the evolution of damage during the fatigue loading, and the formation of delamination between top skin and the core leading to the sandwich structure failure. The presented analytical model presented allows predicting the fatigue endurance of composite sandwich beams using only a limited number of loading levels.

Sandwich panels with rib stiffened and corrugated core have been numerically investigated by Kalnins et al. (2009). Stiffness-based optimization demonstrated significant weight savings over traditional plywood boards. L. He et al. have developed a semi-analytical method suitable for bending analysis of different kinds of sandwich panels (He et al. 2012). They divided the real displacement of sandwich panels into the global displacement field and local displacement field, and determined accurately the real displacement solution and stress distribution of sandwich panels with the help of energy variation principle and the Galerkin approach. Compressive and bending behaviours of wood-based two-dimensional lattice truss core sandwich structures were studied by Jin et al. (2015). The theoretical model and experimental results of the compressive Young's modulus are in good agreement based on the elastic deformation of the dowels. The results have indicated good energy absorption capability of the structure. The debonding of nodes were the primary failure mode of the sandwich structures under bending loads. Atas and Sevim (2010) investigated the impact response of sandwich panels with PVC foam core and balsa wood core. The primary damage modes were found to be the fiber fractures at top and bottom face-sheets, delaminations between adjacent glass–epoxy layers, transverse and in-plane shear fractures of core, and face/core debonding

OBJECTIVE

The main objective of this research is to develop new wood-based sandwich panels with veneer based core reinforcements of different geometries, to optimize the manufacturing technologies, to determine the most important physical and mechanical properties of the newly developed products, to analyze the interrelationship between the materials, technology related parameters and panel characteristics. In this paper, the results of the compression strength tests are introduced.

MATERIAL, METHOD, EQUIPMENT

For samples preparation commercially available beech 3 layered plywood with a thickness of 5mm and polyurethane foam were used. Beech (Fagus sylvatica) sliced veneer sheets were glued using conventional dispersed polyvinyl acetate resin and pressed in cold templates with sinusoidal wave geometry. The same glue was used for veneer tube production; the tubes were prepared by winding the side jointed veneer sheets on a metal tube with a diameter of 60mm. After the resin setting both sine waved sheets and veneer tubes were cut to a width of 50mm and glued to one of the plywood face sheets using a slightly foaming PU glue. The free space of these honeycomb–like cores was filled up with a one component polyurethane foam and covered with the second plywood sheet. Core reinforcements were made in three thicknesses in order to determine the effect of wall thickness on compression strength. The veneer core reinforcements comprised 3, 5 and 7 veneer layers respectively. Panels with the dimensions of 200mmx200mm were prepared containing 9 full tubes and 1,5 long sine waves with the height of in the core. 10 specimens were produced per core type and number of layers. Specimens were tested in compression using a universal Instron testing machine. For the modulus of elasticity determination the cross head displacement was recorded. The measured data was further analyzed using standard statistical methods. Fig. 1 presents the test setup and the veneer reinforcement geometry of the core layer.

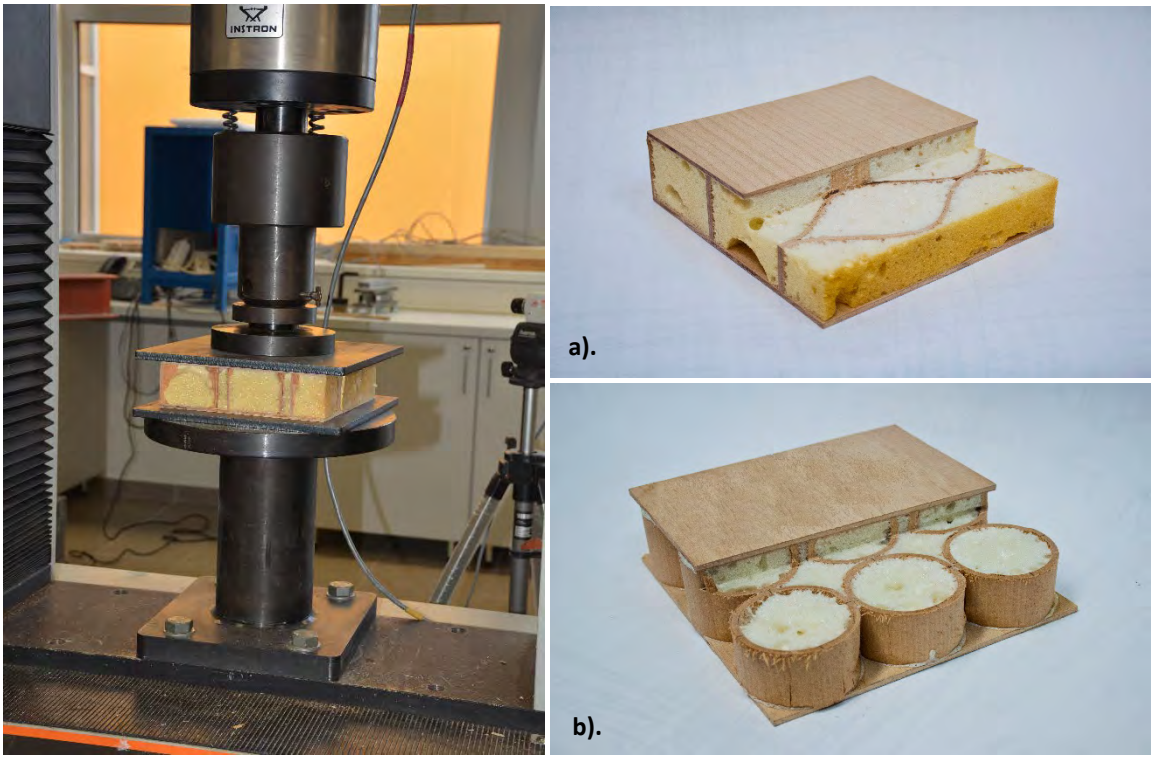

Fig. 1.
The compression test setup and core geometry, a.) sinusoidal reinforcement; b.) tubular reinforcement.

RESULTS AND DISCUSSION

The compression strength and elasticity results are given in Table 1. The positive influence of layer numbers can be observed for both tubular and sinusoidal core geometries and the higher the number of veneer layer the higher the standard deviation. The compression strength of the sandwich panels is significantly higher than similar values of the paper honeycomb core and 8mm thick particleboard skin panels (0,147Mpa) and the sinusoidal core panels are comparable with the cork core agglomerates (Lacreb et al. 2015). Compression load bearing capacity of the tubular core panels exceed multiple times the sinusoidal core panels' similar value (Fig. 2.).

Table 1

		Tubular core			Sinusaiddal core		
		T1	**T2**	**T3**	**S1**	**S2**	**S3**
MOE, Mpa	Mean	121,59	147,67	152,37	20,38	30,28	38,52
	SD	9,83	13,21	19,27	3,83	4,95	4,37
MOR, Mpa	Mean	2,83	5,66	7,29	0,44	0,74	1,04
	SD	0,52	0,62	0,71	0,05	0,10	0,12

The Box plot diagrams of the compression strength and elasticity values are shown in Fig. 2. The seven veneer layer core geometries have the highest values, however the difference between the tubular and sinusoidal core geometries is 6,25MPa in case of MOR and 113,85MPa in case of MOE. The relatively high dispersion of the data underline the imperfections of the core manufacturing technology which should be improved in order to decrease the variability of the cores' mechanical properties. There is linear relationship between number of layers and compression strength and elasticity of the panels, except MOE of the tubular core panels, where the linearity is questionable.

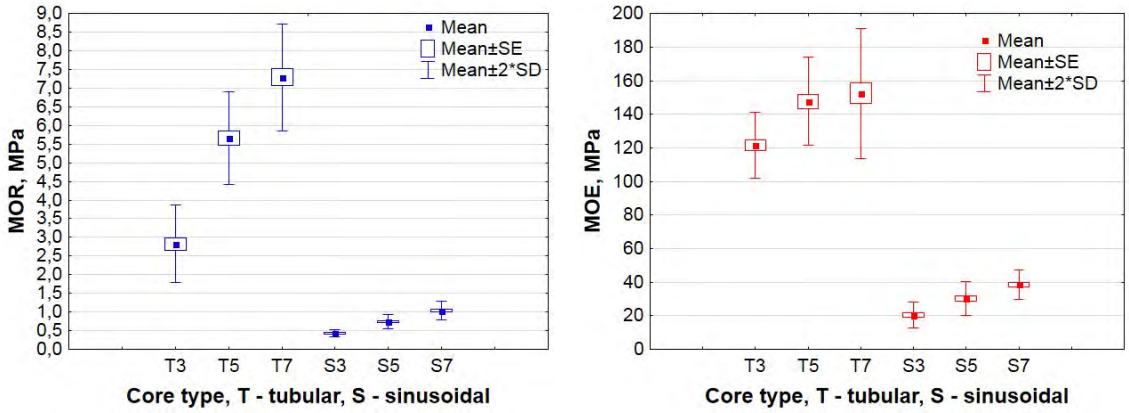

Fig. 2.
The Box and Whisker Plot diagrams of the modulus of rupture and modulus of elasticity in function of core geometry and number of veneer layers.

Fig. 3 shows the scatterplots of MOE and MOR against core types and layer numbers, the linear regression lines and regression equations. The dotted lines represent the 95% confidence regression bands of the mean values. The R square values indicate that the regression models fit well the measured data, except elasticity of the tubular core values, where the residual variability is 58% to the 42% of original variability. In the case of tubular cores the modulus of rupture increase very steeply with the increase of tube wall thickness, in rest the increase is more moderate.

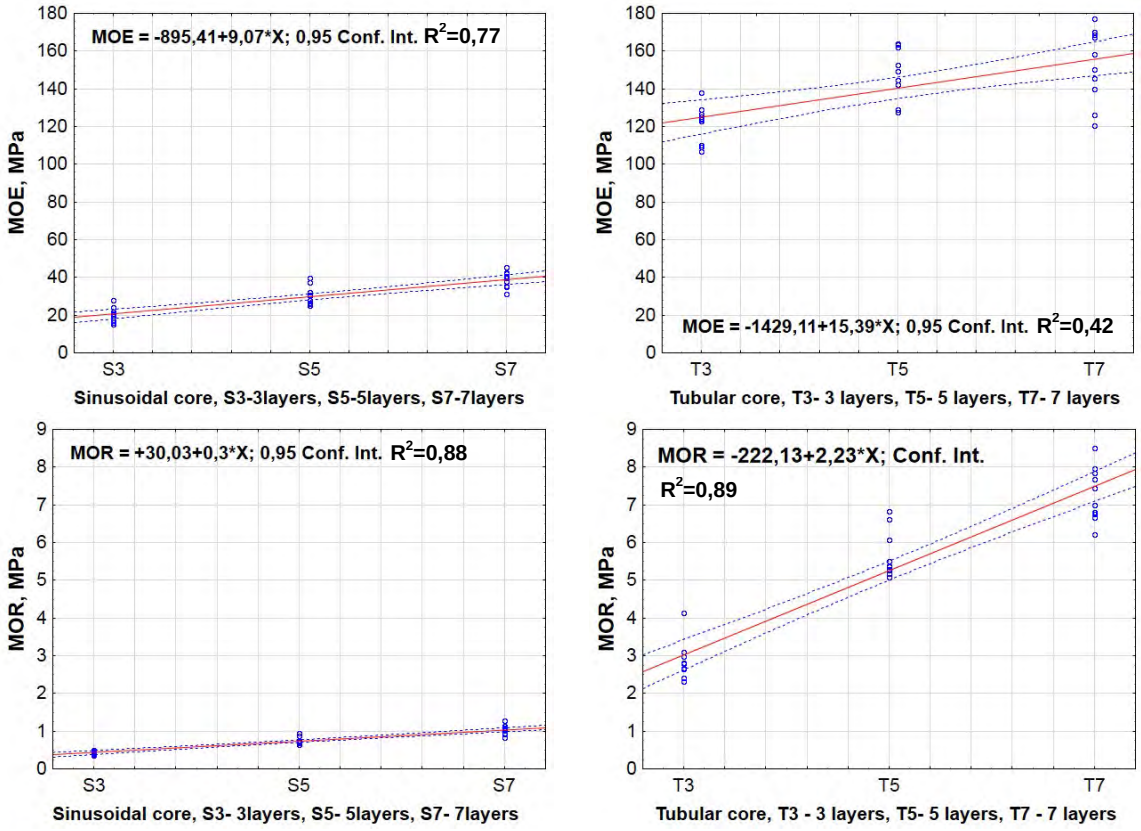

Fig. 3.
Scatter plot diagrams with regression lines of the modulus of rupture and modulus of elasticity in function of core geometry and number of veneer layers.

Typical failure modes are represented in Fig. 4. The buckling failure of the core wall is characteristic for sinusoidal reinforcements, the tubular cores fail in wall crushing and wrinkling.

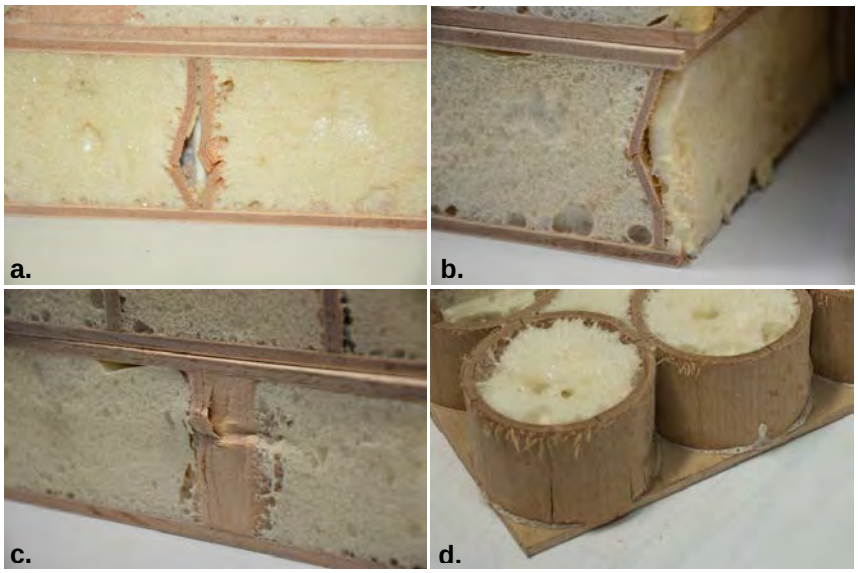

Fig. 4.
Typical failure modes in compression: a, b - buckling failures, c,d – wrinkling failures.

CONCLUSIONS

- The developed veneer reinforcements used in the plywood face, polyurethane core sandwich structures remarkable increase the compression strength and elasticity of the panels
- The strength and stiffness of the tubular reinforcements are 4 to 6 times higher than of sinusoidal reinforcements
- The linear regression models describing the relationship between the number of veneer layers and strength and stiffness fits well on the measured data except modulus of elasticity of the tubular core sandwich panels
- The characteristic failure mode of the tested sandwich structures is the lateral buckling of the reinforcement walls of sinusoidal core and crushing and wrinkling of the tubular walls respectively.

ACKNOWLEDGEMENT

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PHYSICAL AND MECHANICAL PROPERTIES OF TEAKWOOD LVL BONDED WITH EXPANDED POLYSTYRENE

Cláudio DEL MENEZZI
University of Brasília, Faculty of Technology, Dept. Forest Engineering
Campus Darcy Ribeiro, 70910-900 Brasília, Brazil
Tel: 0055 61 3107 0479 E-mail: cmenezzi@unb.br

Ana Paula NAKAMURA
University of Brasília, Faculty of Technology, Dept. Forest Engineering
Campus Darcy Ribeiro, 70910-900 Brasília, Brazil
Tel: 0055 61 2028 7118, E-mail: ana.nakamura@florestal.gov.br

Francis QUEIROZ
University of Brasília, Faculty of Technology, Dept. Forest Engineering
Campus Darcy Ribeiro, 70910-900 Brasília, Brazil
Tel: 0055 61 61 9977 2054. E-mail: francislcq@hotmail.com

Matheus COUTO
University of Brasília, Faculty of Technology, Dept. Forest Engineering
Campus Darcy Ribeiro, 70910-900 Brasília, Brazil
Tel: 0055 61 3349 5336, E-mail: mathescc50@hotmail.com

Abstract

This study presents the physical and mechanical properties of teak LVLs bonded with expanded polystyrene (EPS). The hot-pressing was done at 145°C, 1MPa, for 8min. Three EPS amounts were tested: 96g/m², 192g/m², and 288g/m². We performed the following tests: thickness swelling (TS), water absorption (WA), static bending strength, non-destructive test with stress wave, and glue-line shearing. The panels produced with a amount of 288g/cm^2 presented better results for TS and WA after 2h and 24h. There was no significant difference for static and dynamic elasticity moduli *among treatments. The best treatment was 288g/m² for the modulus of rupture. The stress wave analysis cannot be applied to the composite as a method for predicting mechanical properties. The treatments with 192g/m² and 288g/m² met the minimum requirements for shear strength according to standards EN 314-1 (2004) and ABNT ISO 12466-2 (2012) for dry indoor environments.*

Key words: *nondestructive evaluation; expanded polystyrene; Tectona grandis; wood plastic composite.*

INTRODUCTION

Studies of wood-plastic composites have been carried out since the late 1960s (Rowell et al. 2002). Plastic as a matrix improves the material characteristics, such as resistance to moisture, and insects and fungi attack. Wood particles, such as polymer matrix reinforcement, improve mechanical properties and provide greater composite thermal stability. However, volatiles release, lignocellulosic particle degradation, or even carbonization may occur at temperatures above 200°C (required for the glass transition of some plastics). Therefore, the plastic-wood composites formulations are restricted to thermoplastics with low processing temperatures. Polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), and polystyrene (PS) - with low or moderate crystalline melting temperatures - are among the thermoplastic polymers used for the production of wood-plastic composites (WPCs) (Chindaprasirt et al. 2015).

Polystyrene (PS) is an aromatic polymer made from the styrene monomer, with a glass transition temperature (Tg) of 100°C. Above this temperature, the material gradually liquefies. (Lisperguer et al. 2011). It is characterized by low density, high transparency, and good mechanical properties. It is the third most widely used thermoplastic in industries worldwide, with low production cost and easy processing. In its expanded form (EPS) it is used in packaging and as an insulating material, due to its versatility and dimensional stability. However, after use, it is generally discarded into landfills or incinerated (Poletto et al. 2011).

EPS is a chemically inert, non-biodegradable, but 100% recyclable material. If not recycled, however, it is an environmental problem due to the space occupied by this material in landfills.

(Chagas et al. 2011). Poletto et al. (2011) and Lisperguer et al. (2011) have already investigated the use of polystyrene composites as a matrix reinforced with powder and wood particles. In our previous work (Del Menezzi et al. 2016) it was demonstrated that it is possible to use EPS for bonding wood veneers to manufacture laminated veneer lumber (LVL).

OBJECTIVE

The main goal of this study was to deep our previous work (Del Menezzi et al. 2016) by determining physical and mechanical properties and performs the technical feasibility of the production of *Tectona grandis* LVLs bonded with EPS, with three different amounts of EPS.

METHOD, MATERIALS, AND EQUIPMENT

The study was carried out in Brasília-DF, Brazil, in the Laboratory of Wood Technology, Department of Forest Engineering, University of Brasília, and in the Laboratory of Forest Products of the Brazilian Forest Service. The study used 1.2-mm rotary peeled veneers of teakwood (*Tectona grandis*). They were produced from logs of a commercial forest stand with 13 years of age implanted in the Municipality of Juara/ MT. Lamination was performed with a lathe at Sharewood do Brasil Ltda. Expanded polystyrene (EPS) sheets (Styrofoam® plates) were used as a thermoplastic adhesive. They were available locally, with dimensions of 50x100x0.8cm, and density of 96g/m^3. Three treatments with different EPS amount (96g/m², 192g/m², and 288g/m²) were tested in five replicates, totaling 15 panels. Panels were assembled by inserting *T. grandis* veneers and polystyrene layers, totaling five wood layers and four polystyrene layers. The panel was pressed using Indumec hot-press, at a temperature of 145°C, pressure of 1MPa, for 8 minutes. Panels with the dimensions of 20x20x0.77cm were obtained. The assembled panels were conditioned in an acclimatization room with a relative humidity of 60±5%, and temperature of 20±1°C until constant mass. The following physical and mechanical tests were performed (Table 1):

Table 1

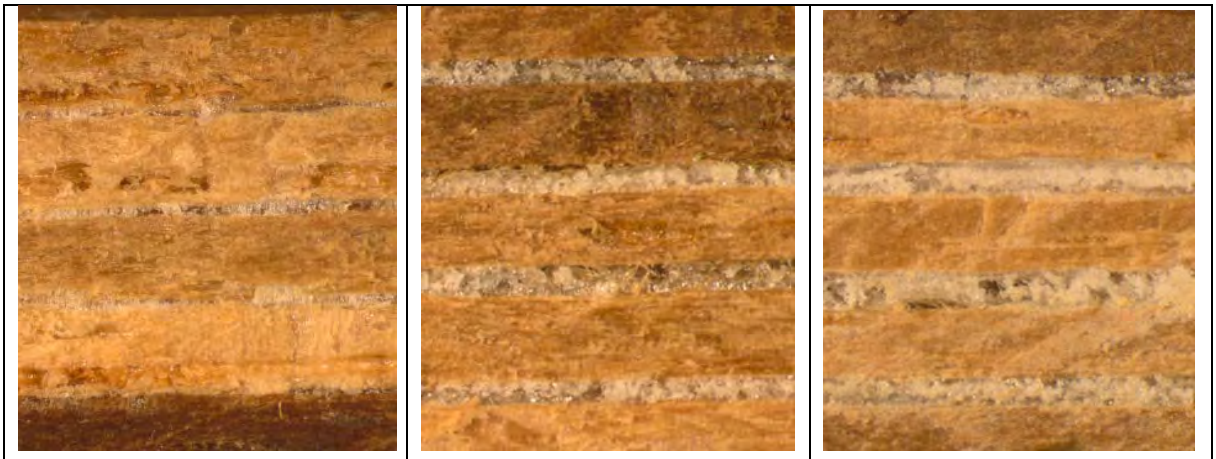
Tests performed for physical and mechanical characterization and applied standards.

Test	Standard
Water absorption (WA) and thickness swelling (TS), 2h and 24h	ASTM D-1037 (2006);
Static flexural strength - Modulus of elasticity (E_M) and Modulus of rupture (f_m), ASTM D-1037 (2006);	ASTM D-1037 (2006);
Stress wave dynamic elastic modulus (E_d)	Del Menezzi et al. (2016);
Shear strength parallel to the glue line - (fv), dry test	EN 314-1 (2004) and ABNT ISO 12466-2 (2012)

The experiment was conducted in a completely randomized design (CRD). Statistical analysis was performed by the ANOVA and Tukey tests, at 5% significance. A simple linear regression model was also generated from joining all the treatments in a single group, correlating E_d with E_M and f_m in order to identify the predictability of the mechanical properties using nondestructive test.

RESULTS AND DISCUSSION

The panels with the three EPS amounts had densities which did not differ significantly from each other. However, the differences between the thicknesses of glue lines in the three treatments are perceptible, as well as the glue line porosity (Fig. 1).



96g/m²	192g/m²,	288g/m²

Fig. 1.
Glue lines of panels produced with different amounts and PS.

The panels produced with of 288g/cm^2 showed better results for WA and TS properties, for both 2 hours and 24 hours (Table 2). There was a tendency of reduction for WA and TS with an increase in OS, possibly due to the hydrophobic characteristics of the thermoplastic material used in the glue.

Table 2
Mean values and standard deviation for apparent density at 12% (ρ), water absorption (WA), and thickness swelling (TS), as a function of EPS amount.

EPS amount	ρ (g/cm³)	WA-2h (%)	WA-24h (%)	IE-2h (%)	IE-24h (%)
0.96 (g/m^2)	0.60a	9.61 (±1.9) a	36.08 (±2.7) a	3.55 (±2.3) a	4.92 (±1.8) a
1.92 (g/m^2)	0.62a	7.97 (±1.0) a	30.26 (±3.9) b	1.95 (±2.7) b	3.84 (±1.4) a
2.88 (g/m^2)	0.59a	5.02 (±0.4) b	25.20 (±2.8) c	0.93 (±1.2) c	1.87 (±0.5) b

Means followed by the same letter in the same column do not differ from each other by the Tukey test at the 5% significance level.

The EPS amounts did not significantly influence the dynamic modulus of elasticity and the static modulus of elasticity (Table 3). The modulus of rupture of the panels produced with the highest EPS amount was superior. Iwakiri et al. (2014) reported higher values for modulus of elasticity (12,450 to 14,274MPa), and similar ones for modulus of rupture (43 to 85MPa) on teak LVL panels bonded with resorcinol-phenol-formaldehyde, polyurethane, and isocyanate.

Table 3
Mean values and standard deviation of mechanical properties of teak LVL bonded with polystyrene as as a function of EPS amount.

EPS amount	E_d (MPa)	E_M (MPa)	f_m (MPa)
96 g/m²	6675.9 (±1466.9) a	8469.8 (±1943.3) a	49.3 (±6.9) b
192 g/m²	7506.5 (±835.6) a	8872.4 (±2172.1) a	53.6 (±12.2) b
288 g/m²	6701.8 (±2206.2) a	9132.6 (±1115.8) a	74.1 (±12.2) a

Means followed by the same letter in the same column do not differ from each other by the Tukey test at the 5% significance level.

The values of modulus of elasticity (E_M) were higher than E_d for all treatments, which is considered atypical for woods. Teles et al. (2011), evaluated the interrelationships between three non-destructive methods and the rigidity in *Sextonia rubra* boards, and Ed was 3.5% higher than Em. Other studies with several species corroborate with the study previously mentioned (Karlinasari et al. 2008; Bucur and Declercq 2006). Nzocou et al. (2006), when analyzing wood-plastic composites with

polyvinyl chloride (PVC), also obtained higher values for E_d (17.6%) in comparison with E_M. Glue line porosity (Fig. 1) may have retarded the wave propagation velocity in the stress wave test, but with less influence on material strength.

The simple linear regression model did not indicate a correlation between the two mechanical properties found in destructive tests with low coefficients of determination (R^2) (Figs. 2 and 3). Nzokou et al. (2006) suggest that the nondestructive stress wave technique may not be appropriate for the prediction of modulus of elasticity in bending of plastic-wood composites.

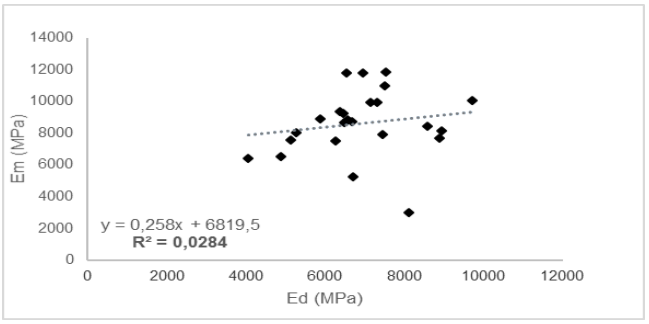


Fig. 2.
Simple linear regression model for Em and Ed.

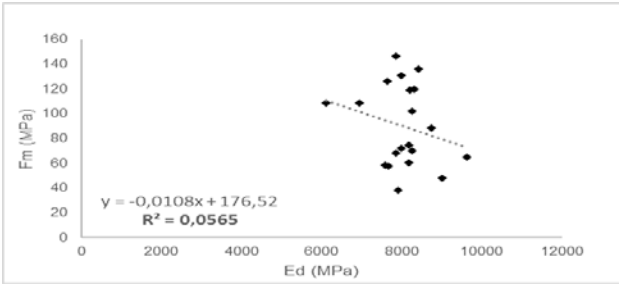


Fig. 3.
Simple linear regression model for Fm and Ed.

The shear strength (fv) (Table 4) of the panels with 288g/m² of polystyrene was the highest; however, the amount of 192g/m² also met the bonding requirements of standards EN 314-1 (2004), and ABNT ISO 12466-2 (2012), in indoor and dry environments.

Table 4

Mean values of the shear strength of LVLs as a function of EPS amount.

EPS amount	Shear strength (MPa)	EN 314-1	ABNT ISO 12466-2
96 g/m²	0.92 (±0.46) b	not met	not met
192 g/m²	1.21 (±0.21) ab	met	met
288 g/m²	1.39 (±0.29) a	met	met

Means followed by the same letter in the same column do not differ by the Tukey test at 5% significance level.
****Parameters referring to class 1 of panels, for indoor and dry environments.***

Lima et al. (2013) studied resorcinol-formaldehyde LVLs (160g/m²) of different compositions of *Pinus oocarpa*, and three Amazonian species with densities similar to those of the present study, and found higher fv values ranging from 4.70MPa to 5.15MPa. Lustosa el al. (2015) produced LVLs bonded with high-density polyethylene and found higher values (2.32MPa). Iwakiri et al. (2010) also obtained higher values (2.05MPa) in LVLs of *Eucalyptus saligna/Schizolobium amazonicum* bonded with phenol formaldehyde. Miranda et al. (2011) verified the presence of 9.2% to 10% of extracts in *T. grandis*, which may have negatively influenced bonding.

CONCLUSIONS

The present study confirms our previous work about the technical feasibility in the production of teakwood LVLs bonded with EPS for indoor and dry environments. The LVLs made with a amount of

288g/m² performed better in the physical and mechanical tests. However, the treatment with 192g/cm² also obtained good results, reaching the minimum plywood quality parameters of standard EN 314-1 (2004). Considering that the latter uses less polystyrene, this amount is recommended for economic reasons.

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DECORATIVE VENEER PROPERTIES OF BUTTERNUT (*Juglans cinerea* L.)

Roman RÉH
Assoc. Prof.Dr.Eng. – TECHNICAL UNIVERSITY in Zvolen, Faculty of Wood Science and Technology
Address: T. G. Masaryka nr. 24, 960 53 Zvolen, Slovak Republic
E-mail: roman.reh@tuzvo.sk

Abstract

Butternut is an introduced species in Central Europe with potential for decorative veneers production and veneering. Decorative veneers with the thicknesses of 0.5, 0.6, and 0.7mm were produced from the raw material with Slovak origin. 10 veneer logs with a length of 140cm and with a diameter of 25–35cm were placed into the water after their transport and consequently the logs were debarked and processed. Veneers were manufactured by off-centre cutting. By means of interrupted off-centre cutting, new and interesting grains and textures of butternut veneer were obtained. Veneers were dried up to a moisture content of 6–8% by drying at a temperature of 100°C. Butternut veneers were subjected to a number of technological test procedures. Butternut as an interesting species for veneer industry is fully recommended. The quality of veneers made from butternut does not differ from the quality of commonly used decorative veneers. Veneer thickness 0.5–0.6mm can be recommended for furniture industry. Glue spread rate of 130–140g·m^{-2} was proposed for particleboard.

Key words: *decorative veneers; biobased composites; veneer properties; butternut; veneering.*

INTRODUCTION

The permanent growth of veneer industries is connected with the reduction of timber supplies of the well-known veneer species (Barbu *et al.* 2014). Therefore the search continues for alternate species, either local or introduced. In screening for new veneer species, it is helpful to know which factors are important for veneer use (Lutz 1971).

This paper includes the recommendation to use the species butternut (*Juglans cinerea* L.) for the production of decorative veneer and veneering. Butternut is an introduced species for Central European wood-processing industry. It is found in the East of North America from southeastern New Brunswick throughout the New England States except for northwest Maine and Cape Cod. The range extends south to include northern New Jersey, western Maryland, Virginia, North Carolina, northwestern South Carolina, northern Georgia, northern Alabama, northern Mississippi, and Arkansas. Westward it is found to central Iowa and central Minnesota. It grows in Wisconsin, Michigan, and northeast into Ontario and Quebec. Through most of its range butternut is not a common tree, and its frequency in North America is declining. The ranges of butternut and black walnut *(Juglans nigra* L.*)* overlap, but butternut occurs farther north and not as far south as black walnut. More detailed information on butternut distribution in Europe is not known. Butternut occurs in parks and in special purpose areas, where its ability can to be included in the assortment of species for the establishment of intensive stands is tested (Soják and Réh 1998).

Common Names: Butternut, white walnut, oilnut
Scientific Name: *Juglans cinerea* L.
Average Dried Weight: 435 kg·m^{-3}
Janka Hardness: 2,180 N
Modulus of Rupture: 55.9 MPa
Elastic Modulus: 8.14 GPa
Crushing Strength: 35.2 MPa
Shrinkage: Radial: 3.4%, Tangential: 6.4%, Volumetric: 10.6% (http://www.wood-database.com/butternut 2017)

Description. Butternut is a hardwood (deciduous) tree growing to 20 m tall, rarely 40 m. It is a slow-growing species, and rarely lives longer than 75 years. It has a 40–80 cm stem diameter, with light gray bark. The leaves are pinnate, 40–70 cm long, with 11–17 leaflets, each leaflet 5–10 cm long and 3–5 cm broad. The whole leaf is downy-pubescent, and a somewhat brighter, yellower green than many other tree leaves.

Heartwood is usually a light to medium tan, sometimes with a reddish tint. Growth rings are darker and form fairly distinct grain patterns. Sapwood is a pale yellowish white. Grain is typically straight, with a medium to coarse texture. Silky natural luster. Semi-ring-porous; medium-large

earlywood pores gradually decreasing to small latewood pores; solitary and radial multiples of 2–3; tyloses occasionally to abundantly present; growth rings distinct; rays barely visible without lens; parenchyma banded (marginal), apotracheal parenchyma diffuse-in-aggregates (sometimes very faint and barely visible even with lens). (Ostry *et al.* 2003) Decay resistance is rated as moderately durable to non-durable; also susceptible to insect attack.

Wood properties. Butternut is easily worked with both hand and machine tools. However, being so soft, butternut has a tendency to leave some fuzzy surfaces after planning or sanding, and sharp cutters and fine-grit sandpaper is recommended. Butternut glues, stains, and finishes well. Butternut has virtually no scent or odor when being worked. Besides the standard health risks associated with any type of wood dust, no further health reactions have been associated with butternut.

Usage. It is available as lumber and carving blanks. Butternut wood is light in weight and takes polish well, and is highly rot resistant, but is much softer than black walnut wood. Oiled, the grain of the wood usually shows much light. It is often used to make furniture, mantelpieces, and interior trims, and is a favorite of woodcarvers along with interior paneling and turnery. It is also suitable for boxes, molding, and crates. It was widely used in churches for detailed woodwork such as intricately carved doors and alters (Nielsen *et al.* 2003). There are not many references to using butternut as plane sliced veneers. The trunks of butternut trees are fluted, which is sometimes still evident in processed lumber - the growth rings in the endgrain may appear more polygonal and faceted rather than perfectly circular. This is not a wood of significant commercial value, but rather a specialty wood.

It normally doesn't leave burn marks and has little dulling effect. The material also works easily with screws, nails, and glue. However, there are some factors to keep in mind. Routing across the grain, for example, can cause the wood to tear out. Although butternut responds well to planning, it's necessary to keep your tools sharp in order to avoid tearing the soft wood. Finally, butternut polishes and finishes beautifully. Because the wood is soft, it's important not to dent it during finishing. Overall, butternut has much to offer.

MATERIALS AND METHODS

Logs (raw material) for this research has Slovak origin and it was taken from Slovak State Forests, the location Levice, Sikenica (Čereš). 10 veneer logs with a length of 140 cm and with a diameter of 25–35 cm were placed into the water after their transport for soaking and consequently the logs were debarked and processed. Veneers were manufactured by the method of off-center cutting in the Development workshops and laboratories of the Technical University in Zvolen (Slovakia). By means of interrupted off-center cutting, new and interesting grains and textures of butternut veneer were obtained. Veneers with the thicknesses of 0.5, 0.6, and 0.7 mm were dried up to a moisture content of 6–8 % by drying at a temperature of 100 °C in the belt (mesh) dryer.

Butternut veneers were subjected to a number of technological test procedures.

Specific Glue Penetration to the Veneered Area

The glue penetration to the veneered area is usually tested on veneer specimen with the dimension of 250 × 300 mm. Common raw particleboard is used for this test and the proper glue amount is investigated (it was tested the glue spread rate from 100 to 220 $g \cdot m^{-2}$).

The evaluation of the amount of glue penetrated to the veneer surface was done with the help of a transparent net with mesh size of 5 × 5 mm. For each value of the glue spread rate 6 specimen had been pressed and the glue penetrating was evaluated in percentage of the total specimen area.

Veneer Adhesion to the Particleboard Substrate (Surface Soundness)

The adhesion between the veneers and the construction material was monitored. The heart of the test lies in the determination of the strength necessary for severing the veneer from the construction material by means of a cylinder made of light metal. For testing, samples of 50 × 50 mm in size were used.

Technological Properties of Veneer from the Aspect of Surface Finish

The technological properties of veneers had been tested for surface finish by transparent paints and systems commonly used for finishing in furniture industry.

As a substrate, for all the tests, three-layer particleboard reversibly veneered 300 × 600 mm; 150 × 300 mm and 100 × 100 mm in size was used.

Transparent coatings were used only. There exist two reasons for the use of final coating material; aesthetics and protection from the end use environment. The esthetics of the final product

varies in many ways, depending upon the selection of the various topcoats available and upon how the final topcoat is handled. The ultimate protection for any wood product finish is dependent upon proper selection of the topcoat for a specific end use.

Three types of clean topcoats were used:

a) Nitrocellulose Lacquers C 1008 and C 1038
b) Synthetic Acid Hardening Lacquer S 1715
c) Nitrocellulose Lacquer Basic C 1026

During surface finishing the panel with veneer was sanded to "knock down" any fibers that have been raised by the application of the finishing material and for further the uniformity of the panel surface.

Specimen were regularly subjected to laboratory tests related to the manufacturing's quality.

Determination the Local Thickness of the Paint. The determination of the local thickness of the paint was done by standards constituting a basis for the evaluation of the paint thickness uniformity on the specimen.

Determination of the Paint Adhesion by Means of the Screen Method (Cross Hatch). The determination of the paint adhesion by means of the screen method was carried out by standards. Adhesion is usually graded in five grades.

Determination of the Resistance to Hot Steam. The intensity of the paint resistance is classified in four grades.

Determination of the Resistance to a Burning Cigarette (Burn Resistance). A burning cigarette was put with about a 10 mm layer of burning ash. The test duration is approximately 60 seconds.

Determination of the Resistance to Chemicals and Selected Consume Liquids (Spot Resistance). The determination of the resistance to chemicals and selected consume liquids was carried out with the drop method.

RESULTS AND DISCUSSION

A survey of the specimen used and their denomination is given in Table 1.

Table 1

Survey and denomination of the specimen for evaluation of the surface finishes

Denomination of Specimen	Substrate	Surface Finish
A	particleboard	C 1008 + C 1038
B	particleboard	S 1715
C	particleboard	C 1026

Specific Glue Penetration to the Veneered Area

Results for specific glue penetration to the veneered area in dependence on the spread rate are given in Table 2.

Table 2

Results of the specific glue penetration to the veneered area in dependence on the spread rate

Veneer Thickness (mm)	Glue Spread Rate ($g \cdot m^{-2}$)												
	100	110	120	130	140	150	160	170	180	190	200	210	220
0.5	-	-	0.033	0.033	0.076	0.090	0.100	0.356	0.436	0.590	0.787	0.990	1.126
0.6	-	-	-	0.014	0.026	0.038	0.188	0.232	0.396	0.580	0.796	0.910	1.222
0.7	-	-	-	-	0.030	0.066	0.117	0.223	0.336	0.397	0.464	0.633	0.728

The test results on glue penetration to the veneered area revealed no substantial glue penetration within the spread rate of 130–140 $g \cdot m^{-2}$, inclusive of followed thicknesses. Butternut from the point of view of glue penetration to the veneered area proved good properties. In actually used

spread rates there is no danger of devaluation of the veneered elements. Glue spread rate of 130–140 $g \cdot m^{-2}$ was proposed for particleboard.

Veneer Adhesion to the Particleboard Substrate (Surface Soundness)

These test results are given in Table 3.

Table 3

Evaluation of veneer adhesion to particleboard substrate [MPa]

Glue Spread Rate ($g \cdot m^{-2}$)	t = 0.5 mm			t = 0.6 mm			t = 0.7 mm		
	Ø	min.	max.	Ø	min.	max.	Ø	min.	max.
100	1.024	0.830	1.202	1.221	1.111	1.321	1.404	0.986	1.802
110	1.264	1.126	1.362	1.263	1.162	1.361	1.566	0.321	1.886
120	1.322	1.143	1.621	1.420	1.221	1.521	1.680	1.399	1.961
130	1.362	1.002	1.543	1.526	1.392	1.643	1.667	1.206	1.896
140	1.403	1.382	1.482	1.630	1.433	1.782	1.702	1.341	2.061
150	1.605	1.503	1.822	1.723	1.701	1.758	1.635	1.218	1.980
160	1.974	1.842	2.202	1.962	1.882	2.121	1.782	1.563	1.913
170	1.707	1.523	1.921	1.720	1.642	1.811	1.708	1.322	2.237
180	1.596	1.423	1.732	1.621	1.532	1.721	1.522	0.980	1.904
190	1.592	1.432	1.648	1.600	1.421	1.681	1.411	0.860	1.786
200	1.329	1.232	1.362	1.426	1.321	1.542	1.545	1.211	1.864
210	1.336	1.321	1.351	1.346	1.212	1.421	1.413	1.012	1.806
220	1.419	1.312	1.542	1.340	1.200	1.366	1.320	0.896	1.523

Test was influenced by the fact that the bottom part of particleboard of the most specimen was destroyed as a result of its imperfectness and not in the glue joint between the veneer and particleboard. We may conclude from this that veneer in the majority of cases kept up with a higher intensity than given in Table 3. The results suggest that adhesion of all three veneer thicknesses of butternut to particleboard substrate highly exceeds required value. Even in lower values of the glue spread rate, veneer adhesion to the substrate was gained with confidence.

Technological Properties of Veneer from the Aspect of Surface Finish

Technological properties of butternut veneer from the aspect of surface finish were studied with a help of the veneer 0.5 mm thick which is a current thickness for decorative veneer and this thickness showed to be a convenient thickness according to the test of specific glue penetration to the veneered area.

Determination the Local Thickness of the Paint. The results of this test are given in Table 4 and represent mean values from four measurements performed at given and mutually comparable places.

Table 4

Evaluation of paint thickness

Specimen	A	B	C
Paint thickness (mm)	0.12	0.11	0.11

The values given in Table 4 correspond to common values at the application of the paint types tested.

Determination of the Paint Adhesion by Means of the Screen Method (Cross Hatch). The results of this test are given in Table 5.

Table 5

Paint adhesion by means of the screen method

Specimen	Adhesion Degree				Resulting Adhesion Degree
A	2	3	2	2	2
B	3	2	2	2	2
C	1	2	1	1	1

As shown in Table 5, all finished types of paints provide adhesion degree 1–2 independently of the type of paint used pointing to excellent or very good properties of butternut veneers with regard to the paint adhesion.

Determination of the Resistance to Hot Steam. The values of paint resistance to hot steam are given in Table 6.

Table 6

Evaluation of the paint resistance to hot steam

Specimen	Type of Injury	Intensity of Injury
A	Blistering, matt gloss, soft surface	3
B	Change of the color shade to white, which was gradually lost, cracking, a more matt and softer surface	2
C	Change of the color shade to white, which was gradually lost, cracking, a more matt and softer surface	2

Resistance to hot steam is purely the matter of coating compositions applied. Hot steam did not affect the quality of butternut veneer.

Determination of the Resistance to a Burning Cigarette (Burn Resistance). The results of this test are given in Table 7.

Table 7

Evaluation of the paint to a burning cigarette

Specimen	Description of the Change of Lacquer	Degree of Damage
A	The paint was burned to the level of veneer, darkened and cracked	3
B	The coating was darkened, its surface was essentially unchanged	2
C	The coating darkened, burned, blistering, delamination of paint	2

It follows from the test of paint resistance to a burning cigarette that all coating composition are less resistant to a burning cigarette.

Determination of the Resistance to Chemicals and Selected Consume Liquids (Spot Resistance). The results of this test are given in Table 8.

Table 8

Evaluation of the paint resistance to chemicals and selected consume liquids

Specimen	*1	*2	*3	*4	*5	*6	*7	*8	*9	*10	*11
	Change Evaluated in Time										
	h d	h d	h d	h d	h d	h d	h d	h d	h d	h d	h d
A	1 1	0 0	0 0	0 0	1 2	0 0	0 0	0 0	0 0	0 0	0 0
B	1 2	0 0	0 0	0 0	1 2	0 0	0 0	0 0	0 0	0 0	0 0
C	1 2	0 0	0 0	0 0	1 1	0 0	0 0	0 0	0 0	0 0	0 0

h – Change evaluated in time of 1 hour
d – Change evaluated in time of 1 day (24 hours)
*1 – shoe polish
*2 – ink
*3 – 10 % CH_3COOH
*4 – 96 % ethylalcohol
*5 – 40 % ethylalcohol
*6 – 10 % citric acid
*7 – sour wild cherry juice
*8 – water
*9 – coffee
*10 – tea
*11 – oil

The resistance of surface finish to chemicals and selected consume matters is in the system under examination a matter of the coating composition quality. All coating compositions used are suitable for surface finish of butternut veneer; no visible changes occurred at using any of the chemicals or consume liquids.

CONCLUSIONS

The popularity of wood veneers has increased significantly worldwide. From the results of tests performed it can be said that butternut as an interesting species for veneer industry and its decorative veneers are fully recommended. The quality of veneer made from butternut does not differ

from the quality of commonly used veneers and the veneer thickness of 0.5–0.6 mm can be recommended for furniture industry. Glue spread rate of 130–140 $g \cdot m^{-2}$ was proposed for particleboard.

Selected introduced woody species suggest good perspectives in the coming years. Future quality of wood and volume production may be secured by the providing of systematic and intense tending of forest stands. Butternut veneer is suitable for all commonly used paint system and all given transparent paint systems may be judged as equivalent and suitable for furniture-making. Butternut is suitable for application in the furniture industry either as a replacement for some commonly used woody species or as a woody species widening the assortment of woody species utilized in furniture industry.

The results obtained suggest that it is possible to recommend its cultivation in larger areas upon properly managed stands (Soják and Réh 1998). It is still necessary to reach more accurate data on the nearest zoning in Central Europe and to realize a research of consumer market in the field of utilizing decorative veneer made of butternut.

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NUMERICAL AND EXPERIMENTAL APPROACH OF BEHAVIOUR OF THE WOOD BASED COMPOSITE SUBJECTED TO CYCLIC BENDING

Mariana Domnica STANCIU
Transilvania University of Brasov, Faculty of Mechanical Engineering
29 Eroilor Blvd, 500036, Romania
E-mail: mariana.stanciu@unitbv.ro

Horațiu TEODORESCU-DRĂGHICESCU
Transilvania University of Brasov, Faculty of Mechanical Engineering
29 Eroilor Blvd, 500036, Romania
E-mail: hteodorescu@yahoo.com

Andrei URSĂRESCU
Transilvania University of Brasov, Faculty of Mechanical Engineering
29 Eroilor Blvd, 500036, Romania
E-mail: ursarescu.andrei@yahoo.com

Pavel Cristian LĂBONȚ
Transilvania University of Brasov, Faculty of Mechanical Engineering
29 Eroilor Blvd, 500036, Romania
E-mail: pavelcristian@live.com

Cornel Sandi BIȚ
Transilvania University of Brasov, Faculty of Mechanical Engineering
29 Eroilor Blvd, 500036, Romania
E-mail: cbit@unitbv.ro

Abstract

Wood mass reduction and the modern tendency concerning a proper use of the lignocellulosic waste have led to the conception of different biocomposite materials with properties similar to those of wood but with an increased dimensional stability. Flaxwood is such a material – a composite material based on wood fibers and polylactic matrix, an alternative to the massive wood, having a homogeneous and an ecological structure, appreciated for its dimensional stability within an environment with humidity variation and being used in musical instruments structure especially.

This paper presents a comparative analysis of the numerical and experimental results for specimens made of flaxwood, subjected to a pulsating cyclic bending. The experimental tests have been performed to determine both the elasto-plastic behavior and the mechanical and structural degradation speed for the material subjected to a cycling bending. Therefore, within the first stage of the study, modulus of elasticity, strain and strength for the specimens under bending, before and after the cyclic loading have been determined. The bending modulus of elasticity has decreased with 8% after 11 cycles of loading, the strength with 5.2% while the strain with 6%. The structural changes in the two phases (before and after the cyclic loading) have been analyzed using SEM. Within the second part of the study the behavior of the lignocellulosic material has been investigated using the finite elements method (FEM), in a hypothesis of an elasto-plastic material (MEP) and of a linear elastic material (MLE). The relative error between the value of the experimental stress and that resulting from the analytic calculus was approximately 0.3%. The best finite elements model from the stresses relative errors point of view (compared with the experimental results) is that of the specimen considered an elasto-plastic material. In such a case the relative error is of approximately 0.8%.

Key words: *wood based composite; pulsating cyclic loading; elasto-plastic behavior; bending; finite elements method.*

INTRODUCTION

Wood is a hygroscopic material that changes its shape and dimensions when exposed to humidity variations, therefore presenting a dimensional instability behavior. This is the reason why the use of wood in musical instruments production especially means to be able to control both the technological parameters which to insure optimal conditions for wood dimensional stability and the environmental conditions during the storage and the usage of the musical instruments. On the other hand it is to be noted that the storage and the usage of the musical instruments depends upon the users which are not always informed about the wood properties (advantages and disadvantages). At present there is an important tendency to replace the wood with wood based composite whose properties to satisfy the dimensional stability requirements. In the future, a nearly complete replacement of wood with this kind of materials has been predicted, the use of massive wood in musical instruments production risking to become a luxury. Modern studies concerning the wood based composites are looking to improve both the mechanical and technological properties and their

long-term viability. During cyclic loading applied on medium density boards (MDF), particleboard (PB), plywood (PL), oriented structural board (OSB), a rapid decrease in fatigue life to a range of 400 to 10 cycles was observed for a stress level of 70% of the average MOR (Bao 1996). In case of composite based on polylactic acid reinforced with cellulose and low density polyethylene filled with cellulose fibres, the incorporation of cellulose into PLA matrix lead to stiffer but slightly more brittle and weaker materials, since Young's modulus increases and tensile strength and elongation at break slightly decrease (Shumigin 2011). Durability of different wood based composites related to adhesive's type was evaluated by cyclic bend stress test (Gaborik et al. 2016). The fatigue life of wood based composite (OSB and plywood) could be predicted by monitoring the energy loss per cycle in a test with several loading cycles, as is mentioned by Sugimoto (2006). Stanciu (2016) noticed that the high tensile strength of the composite is due to reinforcement and type of fibres used, while flexural strength is due to the elastic characteristics close of the two components - matrix and reinforcement, which makes both components to work together. Experimental tests has shown that the failure in a laminated composite is very often progressive in nature, occurring by a process of damage accumulation (Mortazavian 2017; Eftekhari 2016).

Therefore, the problems that arise with these materials concern with the incompatibility at the level of the interface between matrix (PLA) and wood fibers (Oksman 2003; Zhang 2012). Therefore, the fibers percentage has a very important influence on the mechanical properties and on the behavior of lignocellulosic composite materials when subjected to cyclic loadings (Huda 2006).

OBJECTIVE

This paper presents an analysis of the elasto-visco-plastic behavior for a wood based composite material having a polylactic matrix – commercial name: flaxwood, largely used in the field of automotive construction industry, civil engineering, furniture and musical instruments production. This analysis will follow the determination of the elasto-visco-plastic properties of such a composite material in two states (initial state and after subjected to a pulsating cyclic loading in bending). Based on these experimental results a FEM simulation of the material behavior has been done, starting from the hypothesis of a linear elastic material (MLE) and then for an elasto-plastic material (MEP).

MATERIAL, METHOD, EQUIPMENT

Experimental set-up

The flaxwood specimens were cut to a length of 84*mm*, the length of the calibrated portion (distance between the testing machine supports) being of 64*mm*, average width: 10*mm* and average thickness: 7.5*mm*. All these specimens have been divided in categories: first set – composed of 5 specimens, has been subjected to three points bending till rupture, determining the modulus of elasticity in bending, rupture loading, maximum stress and strain. The second set has been tested to 10 pulsating cycles, at a load value representing 70% of rupture load determined for the first set (Fig. 1).

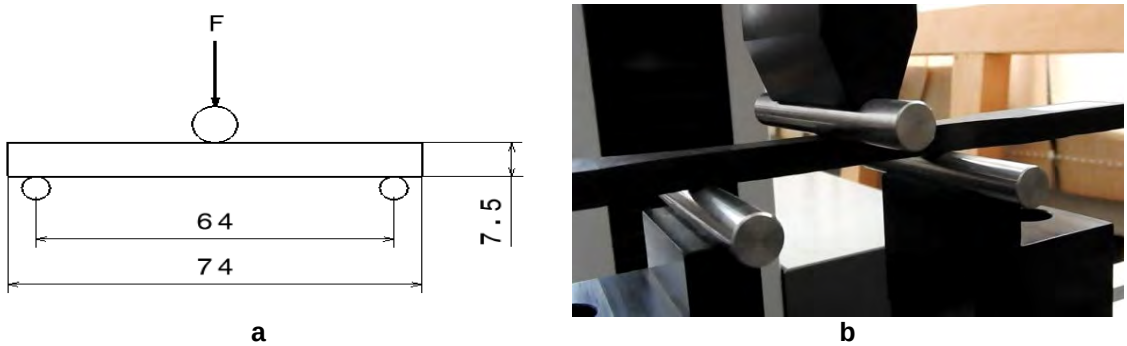

Fig. 1.
LR5K Plus Lloyd's Instruments – Three points bending test of wood based composite (flaxwood): a)The principle of three points bending; b) sample subjected to bending during the test.

During the experimental tests the specimens have been subjected to three points bending, with a velocity of 5 mm/min. These tests have been performed on an universal testing machine type LR5K Plus Lloyd's Instruments (Fig. 1) – owned by the Mechanical Engineering Department – *Transylvania* University of Brasov, present the following characteristics: maximum load domain – 5kN; maximum driving domain: 975mm; load resolution:<0,01% of the used load cell; extension resolution:

<0.1 microns; force cell: XLC-100K-A1; analyzing software: NEXYGEN MT. The experimental results have been collected in electronic format using the software NEXYGEN Plus.

Numerical simulation

To reduce the costs implied by an experimental investigation the finite elements analysis represents a very good solution for the study of a mechanical structure behavior. Therefore, the engineer has to use different computing models that cannot be verified with the experimental tests results or there are not analytical models to compare the results. The main objective of this chapter is to identify the proper mathematical (numerical) model to describe, as precisely as possible, the real behavior of a lignocellulosic composite when subjected to bending. Such a model can be used later to simulate complex mechanical structures made of such a material. The finite elements model – shell type correspond to the real test for a specimen under static bending. Knowing the material function $\sigma = f(\varepsilon)$ geometric and sectional properties and the limit conditions, the recoded loadings during experimental tests are to be applied. Once determined the maximum loaded element, the stresses, displacements and strains will be extracted and compared with the experimental and analytical results. Due to the modeling of the contact conditions the model becomes nonlinear, and the estimated time for resolving the matrix equations increases very much. Therefore, the obtained model represents the best compromise between results accuracy and the necessary time to resolve the equations system.

RESULTS AND DISCUSSION

Three points bending – static loading before and after cyclic stresses

Concerning the behavior of the control specimens and of those subjected to cyclic loading, all under static bending, it is to be observed a decrease of the rupture load with approximately 9% and also a light modification of the material behavior in the elasto-plastic domain, the deformation energy storage capacity being also diminished (Fig. 2, a). Both the strength and the strain under bending have a decreasing tendency with 5-6% (Fig. 2, b). Although the material rigidity does decrease with the increase of the loading cycles, it is to be noted that the lignocellulosic composite material is stable from the mechanical point of view, the characteristic stress-strain curve having the same mathematical expression, both before and after the cyclic loading (Fig. 2, b). The composite material analyzed in the two phases has shown a brittle rupture, as it may be observed in Figure 2, a and b.

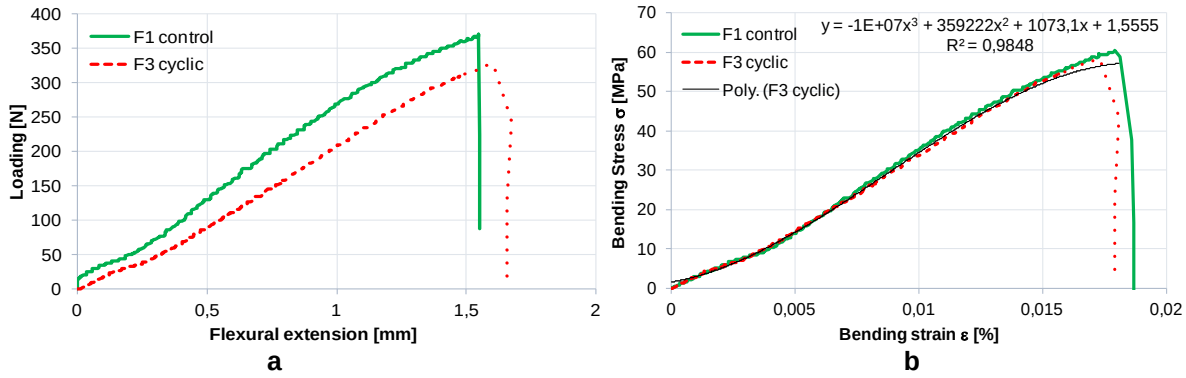

Fig. 2.
Load-displacement (a) and stress-strain (b) functions under static bending.

From all specimens subjected to cyclic loadings, a percentage of 80% did breaking during the loading cycles. Only the remaining ones could be tested to static bending. Therefore, the average value of the modulus of elasticity in bending has decreased with approximately 8% while the flexural rigidity with approximately 12% (Fig. 3, a and b). It is very important to know about this tendency when using a wood based composite for musical instruments structure where flaxwood components are subjected to cyclic loadings with a relatively reduced load intensity.

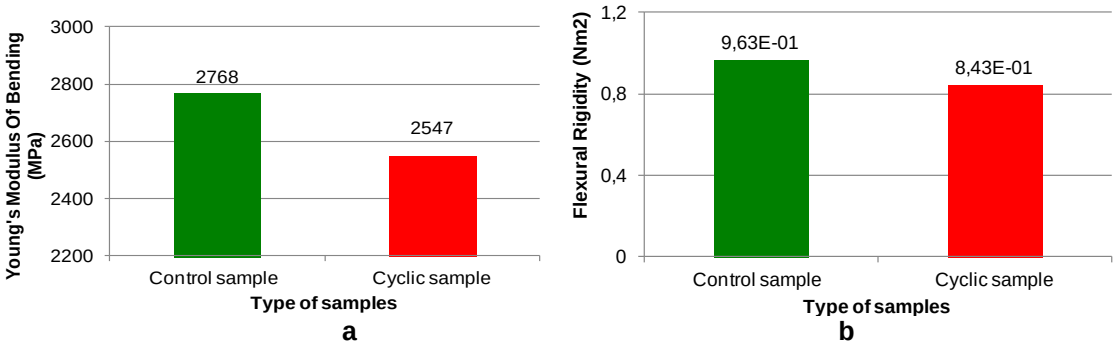

Fig. 3.
a) Variation of the modulus of elasticity; b) variation of flexural rigidity in case of control specimens and of those under a cyclic loading

Three points bending – cyclic loading

In case of pulsating cyclic loadings the specimens have been periodically loaded with 250N, load variation speed of 5mm/min, during 11 cycles. From Fig. 4 it is to be noted that the stress has approximately equal values for each pulsating cycle, compared with the strain whose tendency is to increase with the number of cycles. The plastic strain recorded after the first loading cycle is of 0.09% (Fig. 5, a) while the final strain value (after 11 loading cycles) is of approximately 0.18%. So, 50% of plastic deformation has been obtained after the first loading cycle while, during the next cycles, a reduction of the deformation rate took place according to equation from Fig. 5, b.

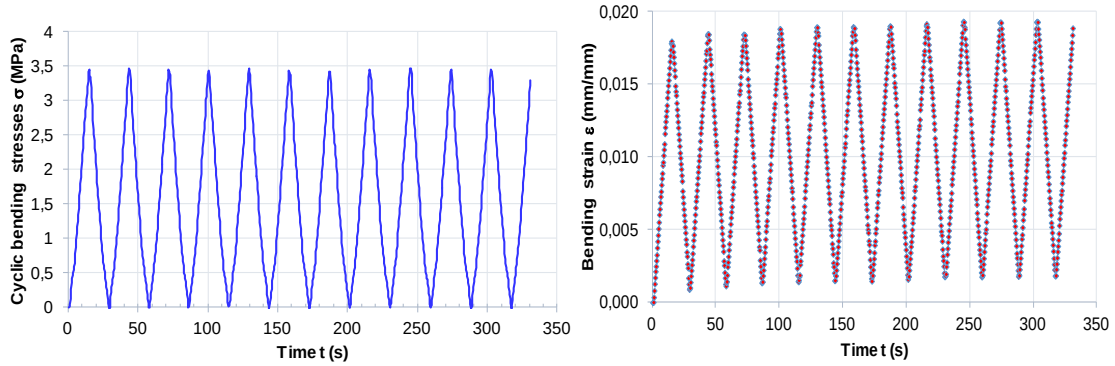

Fig. 4.
Cyclic variation of bending stress (a) and (b) strain.

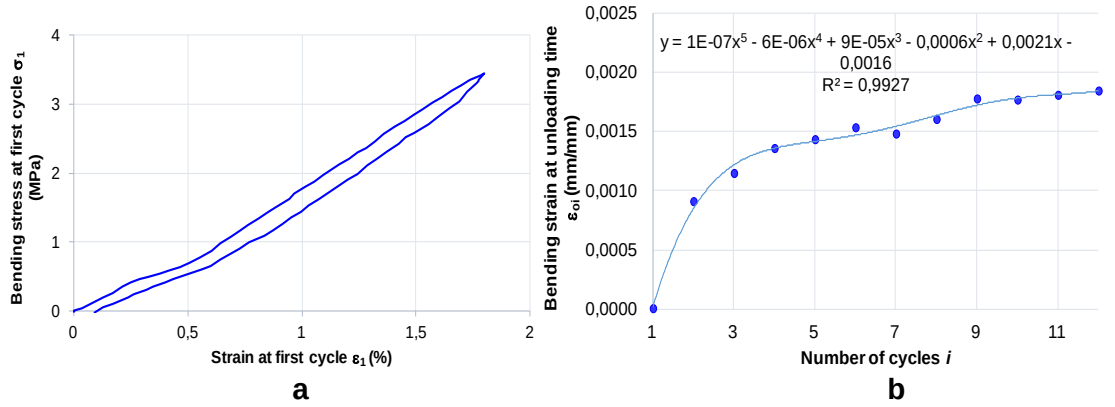

Fig. 5.
Strain variation in the plastic domain: a) after the first loading cycle; b) for each loading cycle.

SEM analysis

During the investigating research the analysis has been performed using a scanning electron microscope - Hitachi S-3400N type II. This analysis has been concentrated on specimens surfaces topographic study in the section where fracture took place under static bending and after a cyclic loading. In Fig. 6 the topographic images of the stretched area and compressed area have been represented.

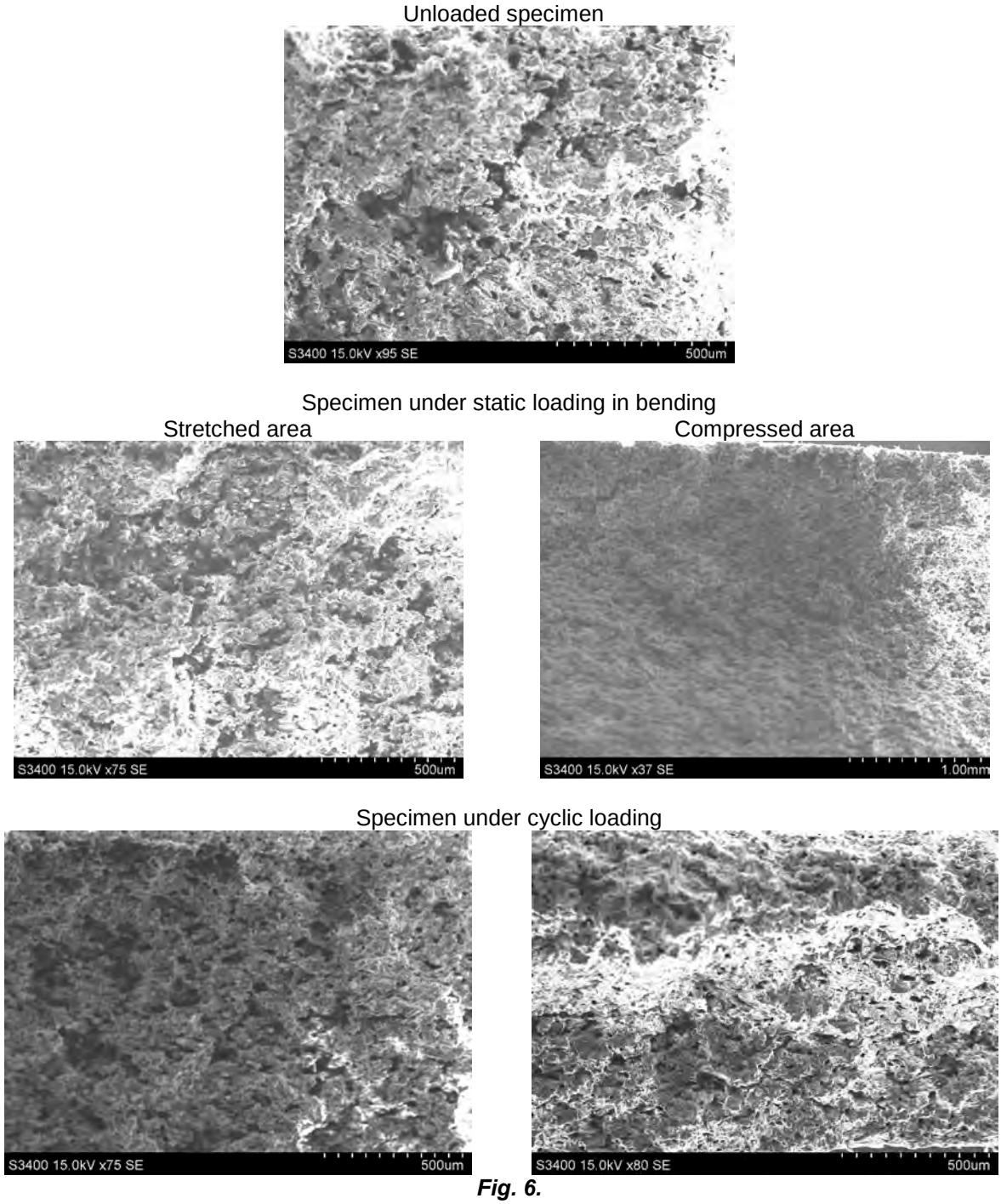

Fig. 6.
Lignocellulosic composite surface topographic analysis in the section where specimens fracture took place.

Structural modifications due to the normal stresses variation in section are mainly present within the compressed area where the structure becomes compact. For the stretched area the elements insuring the strength in tension are represented by the material lignocellulosic fibers. If comparing the compressed surface topography under static bending with that of under pulsating cyclic

bending it is to be observed that these are different: more compact and homogeneous in the first case, the strength being insured by the matrix and nonhomogeneous in case of cyclic loading where a fibers dislocation in the matrix takes place during specimens upload-download (Fig. 6).

Cyclic loading behavior FEM analysis

Within the first phase the lignocellulosic composite material has been modeled in two hypostases: linear–elastic material (denoted by MLE) and elasto-plastic material (denoted by MEP). Fig. 7 presents the two material curves, used for material modeling, and the experimental curve. The linear-elastic material characteristic curve represented in Fig. 7, has the same slope with that experimentally determinated. It follows that the modulus of elasticity values are also equal.

To determine the elasto-plastic material characteristic curve using FEA it has been necessary to introduce in the computing program the value of : real stress (σ_r), real strain (ε_r) and plastic strain (ε_p). This modeling has been applied to the stress (and strain) values greater than 11MPa – considered the material elasticity limit according to the characteristic curve experimentally determinated – Fig. 7. In the stress-strain function graphical representation (Fig. 7) one can observe that the elasto-plastic modeling approximates much better the real behavior compared with the linear-elastic modeling, both for stresses and for strains. The relative error is approximately 0.8% in case of stresses and 5% in case of displacements. Even if the maximum stress obtained through a linear – elastic modeling is of 60MPa (witness specimen rupture stress value approximately) it is to be observed that the strain value at rupture is with 27% greater than that experimentally determinated.

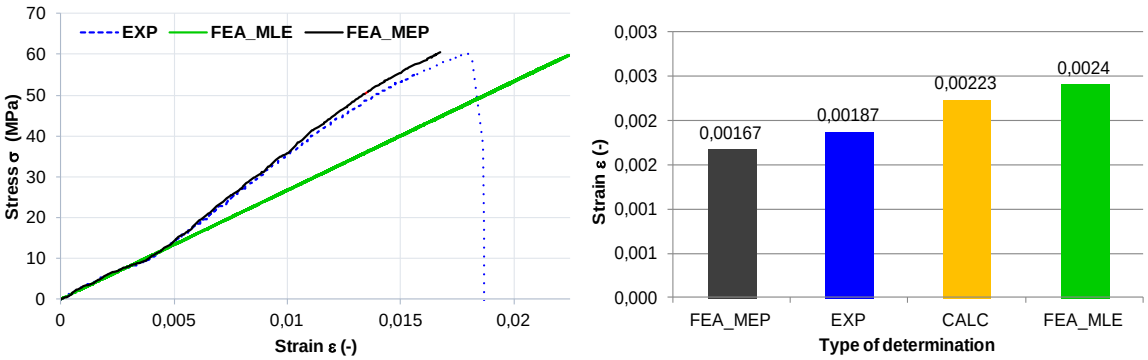

Fig. 7.
Experimentally determinated characteristic curves – FEA (a); Strain relative error variation (b) (Exp – experimentally curve; FEA_MLE – characteristic curve for a linear – elastic material; FEA_MEP- characteristic curve for an elasto-plastic material; CALC – analytically computed value).

After the finite elements model processing, the main quantities of interest were: stresses, displacements and strains, in the most loaded nodes of structure. In Fig. 8 the stresses and displacements distribution have been represented, in case of a specimen modelated with an elasto-plastic material and also the maximum point of these values.

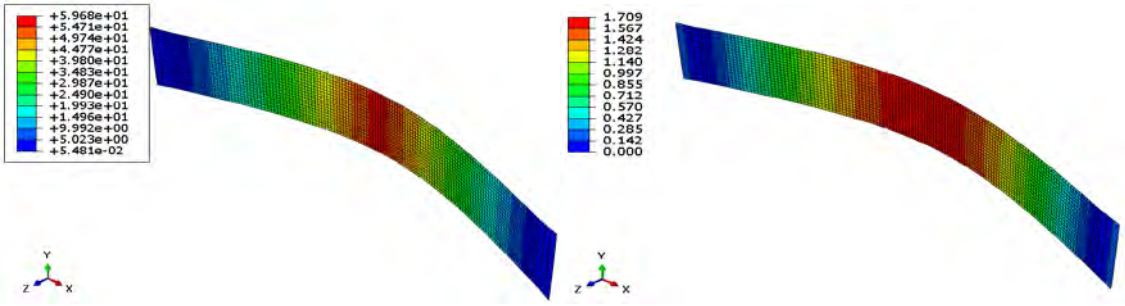

Fig. 8.
Displacements and stresses distribution in case of the modeling of an elasto-plastic material.

In case of the cyclic loading modeling, the results obtained have been represented in Fig. 9, where one can comparative observe the strain variation in time for the maximum loaded node.

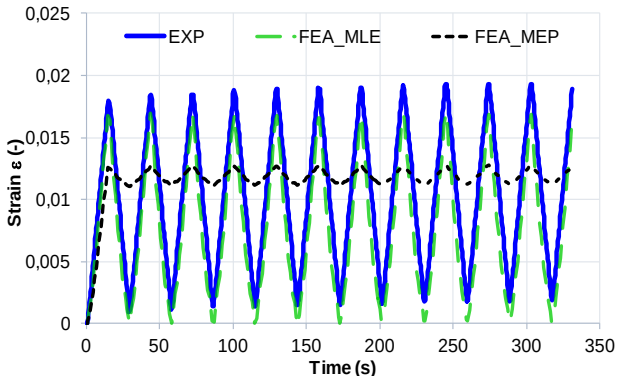

Fig. 9.
Strain variation - experimental and numerical determinations.

In contrast to the simulation of the behavior under static bending, in which the elasto-plastic model does show the real behavior of the composite material, in case of cyclic loadings one can observe an overlapping of the linear-elastic model behavior with that of the real material. This is the reason why it may be concluded that the numerical analysis of lignocellulosic composite structures implies the adequacy of the material mathematical models to the type of loading. From static loadings FEA analysis the material model corresponding to the real behavior is an elasto-plastic one while, in case of dynamic loadings, the proper model is linear-elastic, with an increment applied to the modulus of elasticity to simulate the viscous behavior.

CONCLUSIONS

This paper presents an analysis of the lignocellulosic composite materials under cyclic loadings, the study being concentrated on a commercial material – flaxwood. This analysis has been performed through experimental and numerical investigations.
The results obtained have highlighted the following aspects:

- the investigated lignocellulosic composite presents a low strength under bending but a relatively high rigidity compared with other materials, the strain being of 0.0187%;
- specimens rupture mode indicated a brittle behavior;
- under cyclic loadings, the material capacity to store deformation energy decreases with 50%, even after the first loading cycle, and more than 80% of tested specimens showed a sudden decrease of strength under cyclic bending, through their rupture after the fourth and the fifth loading cycles;
- during the cyclic loadings structural changes occur, represented by the damage of the interface matrix-wood fibers that, finally, lead to the material rupture, (Fig. 6);
- in case of the numerical modeling the "reverse engineering" method has been chosen for simulation, interpolating the experimental data with theoretical data, resulting two distinct mathematical models according to the loading type: static (elasto-plastic model) or dynamic (linear-elastic model).

ACKNOWLEDGEMENT

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INDUSTRIAL RESEARCH CONCERNING THE IMPROVEMENT OF SOME PHYSICAL AND MECHANICAL PROPERTIES OF HDF PANELS USED AS DOORSKINS

Nicolae BĂDIN
KASTAMONU Romania
Str. Ierbuş nr. 37, 545300 Reghin, Romania
E-mail: nicolae.badin@kastamonu.ro

Mihaela CÂMPEAN
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: campean@unitbv.ro

Abstract

The present research envisaged the investigation of several solutions in order to improve some physical and mechanical properties of 3mm thin HDF panels used as doorskins. Six different composition recipes were applied, by varying the percentual participation of hardwood / softwood fibers and bark. The density, surface absorption, bending strength, modulus of elasticity and internal bond of the HDF panels manufactured on an industrial line were determined and the best performances were obtained for the recipe with 20% hardwood fibers/ 80% softwood fibers and less than 5% bark in the composition recipe. Hereinafter, the influence of spraying the fiber mat before pressing, by means of water and two different release agents, was also tested. The obtained results are applicable at any HDF producer, and can be used for process optimization.

Key words: *high-density fiberboards; density; surface absorption; bending strength; modulus of elasticity, internal bond; composition recipe; release agent; process optimization.*

INTRODUCTION

High-density fiberboard (HDF) (Fig. 1) is an engineered wood product produced from fine ligno-cellulosic fibers, combined with a synthetic resin and joined together under high pressure and heat, to form panels (Irle and Barbu 2010). HDF is a non-load-bearing product for interior use in dry conditions, as its mechanical strengths are modest (Table 1) and it is not moisture-resistant. It is produced in a wide range of thicknesses (from 2 to 25mm). The thinner panels, as the ones manufactured by KASTAMONU, are used as doorskins (Fig. 2), backs for cupboards, wall claddings and interior decorations for cars.

The main advantages of this material are its high stability, the dense and smooth surfaces, as well as its "woody" aspect. HDF is suitable for different coatings (veneering, laminating, painting, varnishing), being very appreciated for interior design applications due to easy processing and versatile shapes and colors.

Fig. 1.
Raw high-density fiberboards.

Fig. 2.
Use of high-density fiberboard as doorskin.

Table 1

Standard requirements for main HDF properties (EN 622-5:2009)

Ref.N°	Property	Unit of measure	Value
1	Moisture content	%	4 - 11
2	Density	kg/m^3	>700
3	Density deviation	%	± 7.0
4	Thickness swelling, 24h	%	For 2.5 – 4 mm: ≤35 For 4 – 6 mm: ≤30 For 6 – 9 mm: ≤ 17
5	Internal bond	N/mm^2	≥ 0.65
6	Bending strength	N/mm^2	≥ 23
7	Modulus of elasticity	N/mm^2	≥ 2700
8	Surface absorption	mm	>150
9	Formaldehyde emission	mg/100g	≤ 8 (for direct indoor use)(E1)

The main properties of HDF panels used as doorskins are: density, surface absorption, bending strength, modulus of elasticity and internal bond.

The density of HDF significantly influences the mechanical properties, so the general trend is to attain a density as high as possible (around $1000kg/m^3$), but without affecting the costs of the product. Optimization is possible by varying the composition recipe of raw materials (percentual participation of softwood fibers / hardwood fibers / bark).

The surface absorption is the second important physical property of HDF panels. It has a significant influence upon the gluing and varnishing quality, but also on the product costs. Thus, a high surface absorption increases quality, but also the consumptions and thus the costs, while a too low surface absorption affects the quality by lowering the consumptions. The ideal (envisaged) situation is a clearly differentiated surface absorption on the face and on the back of the panel. Thus, the surface absorption on the back should be higher, in order to achieve a good gluing on the door frame, while the surface absorption on the face should be lower in order to reduce the varnish consumption and thus the product costs. Optimization is possible by using different filling mixtures (Danuta and Monder 2015), but also by using release agents to wet the fiber mat before it enters the press, or by varying some pressing parameters (*e.g.* the closing time).

The bending strength represents the most important mechanical property of any wood-based panel, as it characterizes its consistency and the ability to stand external factors of stress and destabilization. An interesting comparison between the bending strength of MDF and HDF panels made from different fast-growing species is provided by Alpar ***et al.*** (2010).

The modulus of elasticity in bending characterizes the ability of the HDF doorskin to adhere to the shape of the door frame, even if this contains 3D-mouldings, and therefore it is of outmost importance. The value of the modulus of elasticity should be at least $3500N/mm^2$ to provide proper stability and strength.

The internal bond represents another important mechanical feature of the HDF panel, which characterizes the gluing quality and also the property-uniformity of the panel in different directions and planes. It is directly influenced by the panel density and by the accuracy of the glue-spreading process. The closing time of the press is also important: this has to be as short as possible, in order to enhance the formation of solid crystalline bonds between the fibers.

OBJECTIVE

The main objective of the present research was to test different composition recipies of HDF by varying the percentual participation of hardwood / softwood fibers and bark in the composition recipe, as well as by using different release agents during the mat formation. The final outcome was to explore the effects of these measures upon several physical and mechanical properties of thin HDF and upon the overall quality of the product.

MATERIAL, METHOD, EQUIPMENT

The material used within the present research consisted of 3mm thick raw high-density fiberboards (HDF), manufactured on the industrial line at KASTAMONU ROMANIA.

Six combinations resulted by varying the percentual participation of hardwood / softwood fibers in three variants (20/80; 30/70; 40/60) and the percentual participation of bark in two variants (below and above 5%).

A number of around 7000 pcs. of HDF panels were produced from each recipe. The pressing parameters were: temperature *T*=150°C; pressing time *t*=30s; pressure *p*=180bar; press closing time $t_{closing}$=2s.

In order to test also the influence of a release agent sprayed over the panel face before pressing, three alternatives were investigated: water; the release agent type PAT® 2003 XE by Würtz (Germany), diluted in water at a concentration of ca. 4% and the release agent type MOULEX WE07BSP by ADDITEK (France), also diluted in water.

After pressing, the boards were conditioned for 30 minutes at ambiental temperature and then, specific test pieces were cut out of eight randomly selected panels from each recipe, according to EN 326-1:1994 (confirmed 2014), in order to determine the most relevant physical and mechanical properties of HDF boards used as doorskins: the density, the surface absorption, the bending strength, the modulus of elasticity in bending and the internal bond.

Density

The HDF density was determined according to EN 323:1996. Sixteen test pieces from each of the eight test-panels from each recipe, sized at 100mm x 100mm x 3mm, were first weighed at an accuracy of 0.01g. The thickness (*t*) was measured in the central point by means of a micrometer at an accuracy of 0.01mm and the dimensions b_1 and b_2 were measured at mid width and length, according to Fig. 3, by means of a sliding gauge at an accuracy of 0.1mm.

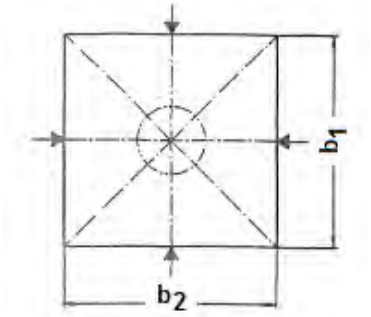


Fig. 3.
Measuring the dimensions of the test pieces for HDF density determination.

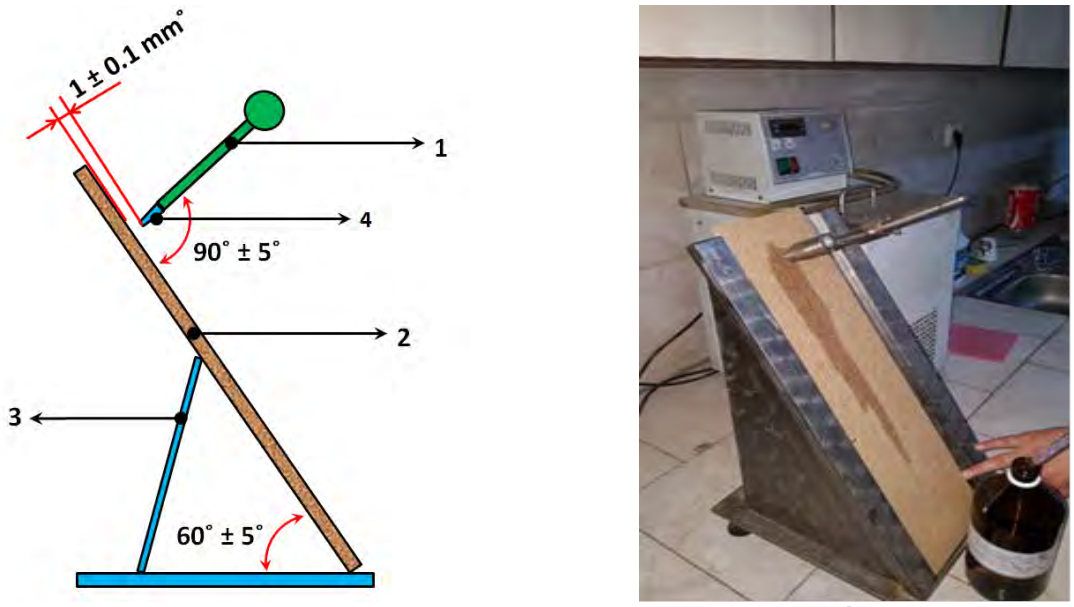
The density of each test piece was then calculated according to the formula:

$$\rho = \frac{m}{b_1 \cdot b_2 \cdot t} \cdot 10^6 [kg / m^3] \quad (1)$$

where: *m* is the sample mass, in g;
b_1, b_2 – sample dimensions, in mm;
t – sample thickness, in mm.

Surface Absorption

The surface absorption was determined according to EN 382-1:1993. Three test pieces from each of the eight test-panels from each recipe, sized at (100±2)mm x (500±2)mm x 3mm were placed on a (60±5)° inclined support (Fig. 4). The pipette for dropping the toluene solution was placed at a distance of 1±0.1mm, perpendicular to the panel surface. 1 ml of toluene was dropped twice on the panel surface at a time interval of 4s and left to flow. The maximum length of the trace was measured along a line parallel to the test piece margins, at an accuracy of ±1mm. The operation was repeated on the other panel face as well. The values obtained, represented the surface absorption (*As*), in mm, with differentiated values on the panel face and on the panel back, respectively.



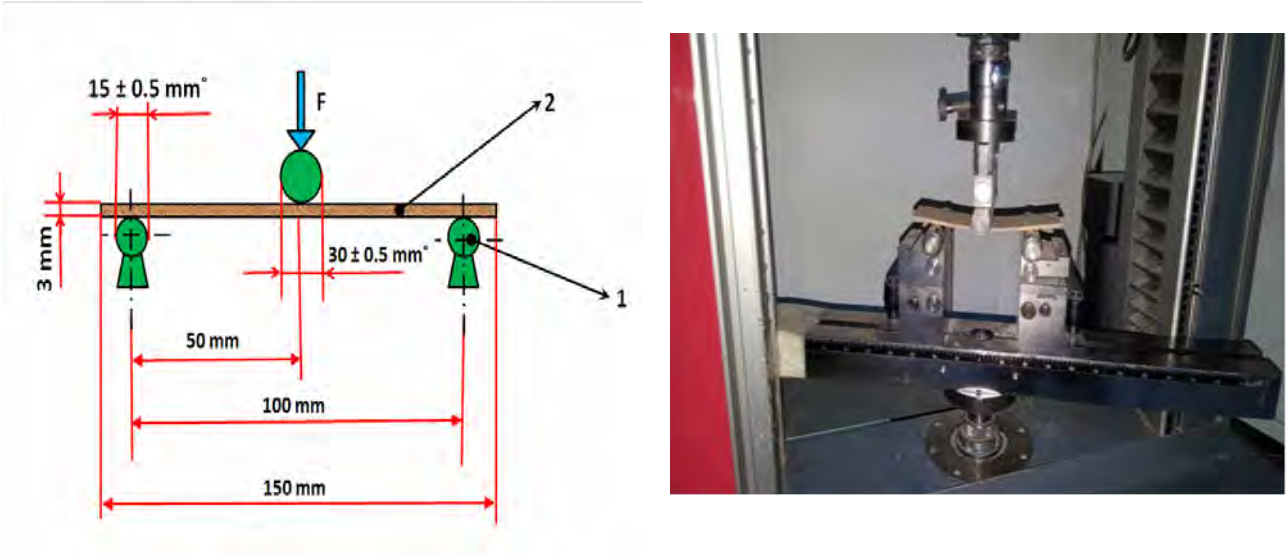
a. b.

Fig. 4.
Determination of the surface absorption of HDF panels:
a-principle, with 1-pipette; 2-test piece; 3-inclined support; b-expermental set-up.

Bending Strength and Modulus of Elasticity in Bending

The bending strength and modulus of elasticity in bending were determined according to EN 310:1993 (Fig. 5a). Six samples from each of the eight test-panels from each recipe, sized at 150mm x 50mm x 3mm, were tested by means of the ZWICK Z010 equipment (Fig. 5b).

The distance between the centres of the supports was adjusted at 100mm. The test piece was placed flat on the supports, with its longitudinal axis at right angles to those of the supports with the centre point under the load, as shown in Fig. 5a. The load was applied at a constant rate of the cross-head movement throughout the test. The rate of loading was adjusted so that the maximum load was reached within (60±30)s. The deflection in the middle of the test piece (below the loading head) was measured to an accuracy of 0.1mm. These values were plotted against the corresponding loads measured to an accuracy of 1% of the measured value. The maximum load was recorded to an accuracy of 1% of the measured value.



a. b.

Fig. 5.
Determination of the bending strength and modulus elasticity in bending of HDF panels: a-principle, with 1-supports; 2-test piece; F-loading force; b-ZWICK 010 testing machine.

The bending strength of each test piece (f_m) was calculated according to the formula:

$$f_m = \frac{3 \cdot F_{\max} \cdot l_1}{2 \cdot b \cdot t^2} [N/mm^2] \quad (2)$$

where: F_{max} is the maximum load, in N;
l_1 – distance between the centres of the supports, in mm;
b – test piece width, in mm;
t – test piece thickness, in mm.

The modulus of elasticity in bending (E_m) was calculated according to the formula:

$$E_m = \frac{l_1^2 \cdot (F_2 - F_1)}{4 \cdot b \cdot t^2 \cdot (a_2 - a_1)} [N/mm^2] \quad (3)$$

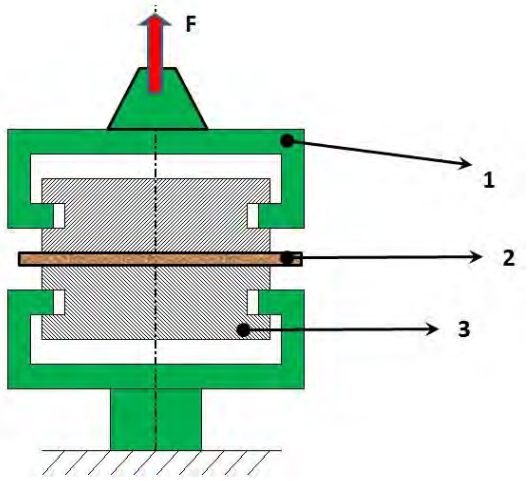
where: $(F_2 - F_1)$ is the increment of load on the straight line portion of the load-deflection curve;
$(a_2 - a_1)$ – deflection increment at the mid length of the test piece corresponding to $(F_2 - F_1)$.

Internal Bond

The internal bond was determined according to EN 319:1993 (Fig. 6a). Four samples from each of the eight test-panels from each recipe, sized at 50mm x 50mm x 3mm, were used. Each sample was first glued to the metallic jig by means of a melting adhesive (Fig. 6b) and then subjected to a tensile force by means of a ZWICK Z010 equipment (Fig. 6c) until rupture occurred. The maximum force which produced failure was recorded, and then, the internal bond was calculated according to the formula:

$$f_{t1} = \frac{F_{\max}}{a \cdot b} [N/mm^2] \quad (4)$$

where : F_{max} is the maximum load, in N;
a, b – length and width of the test piece, in mm



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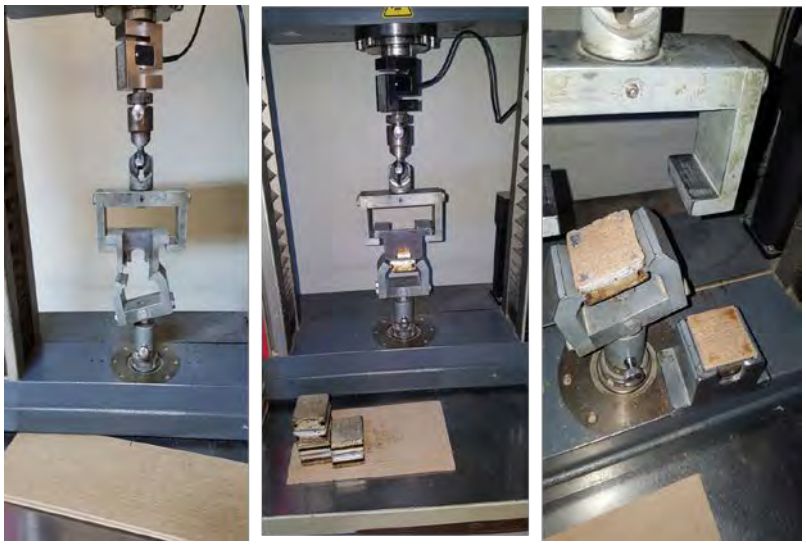


c.

Fig. 6. Determination of the internal bond of HDF panels:
a - principle: 1 – clamping device; 2 - test piece; 3 – metallic jig; F-load (tensile force);
b – gluing of wooden sample on metallic jig;
c - ZWICK 010 testing machine.

In order to compare the results, the mean and the standard deviation from all values obtained for each property and each recipe were calculated.

RESULTS AND DISCUSSION

Influence of the Composition Recipe Upon the Physical and Mechanical Properties of HDF Panels

The results concerning the influence of the percentual participation of hardwood / softwood fibers and bark in the composition recipe of HDF panels upon the properties of HDF panels are presented in Table 2.

Table 2

Influence of the percentual participation of hardwood / softwood fibers and bark upon the properties of 3mm thick HDF panels – mean values and (standard deviations)

Percentual participation in the raw material recipe		Density,	Surface absorption on: face,	back,	Bending strength,	Modulus of elasticity,	Internal bond,
Hardwood/softwood fibers, %	Bark, %	kg/m^3	mm	mm	N/mm^2	N/mm^2	N/mm^2
20/80	≤5	1078 (26.55)	446 (38.62)	398 (34.68)	79.60 (3.28)	6806 (196.84)	2.52 (1.31)
	>5	1050 (21.70)	420 (37.75)	355 (30.36)	71.21 (2.20)	5583 (82.87)	1.74 (0.92)
30/70	≤5	1063 (20.39)	405 (18.02)	310 (18.02)	77.74 (3.39)	6223 (377.95)	2.04 (1.06)
	>5	955 (23.19)	263 (6.81)	217 (31.01)	62.57 (4.01)	5513 (377.99)	1.78 (0.95)
40/60	≤5	1025 (28.63)	337 (17.06)	227 (7.55)	68.96 (4.66)	4896 (355.85)	2.08 (1.07)
	>5	935 (29.40)	229 (14.11)	205 (10.00)	64.86 (4.88)	4926 (170.96)	1.60 (0.83)

By comparing these values to the standard requirements given in Table 1, one may notice that all values comply with these requirements and even exceed them by far.

However, the main aim of the present reserach was an in-deep investigation concerning the possibility to achieve optimum effects by varying the composition recipe of the panels. Thus, it was

established that reducing the percentual participation of softwood fibers in favor of the hardwood (beech) fibers in the composition recipe of HDF doorskins from 80% to 60% (Fig. 7) leads to the:

- decrease by 4.9% of the density in case of bark content ≤5% and to a decrease by 10.9% of the same, when the bark content was >5%;
- decrease by 24.4% of the surface absorption on the face in case of bark content ≤5% and to a decrease by 43% of the same, when the bark content was >5%;
- a decrease by 45.5% of the surface absorption on the back in case of bark content ≤5% and to a decrease by 42.2% of the same, when the bark content was >5%;
- decrease by 13.4% of the bending strength in case of bark content ≤5% and to a decrease by 8.9% of the same, when the bark content was >5%;
- decrease by 28.1% of the.modulus of elasticity in bending in case of bark content ≤5% and to a decrease by 11.7% of the same, when the bark content was >5%;
- decrease by 17.5% of the internal bond in case of bark content ≤5% and to a decrease by 8% of the same, when the bark content was >5%.

These results show that the increase of the beech fibers participation in the recipe does not have benefitting effects upon the panel density and the related mechanical properties. The best results were obtained for the recipes with the minimum percentage (20%) of hardwood fibers, both in the case with lower bark amount (≤5%) and with higher bark amount (>5%).

As far as the influence of the percentual participation of bark is concerned, the results clearly show that all recorded values are lower for the panels with more bark: they are less dense, less absorptive, weaker, less elastic and the gluing is poorer.

Thus, according to the obtained results, the composition recipe which allows the best mechanical performances (bending strength, modulus of elasticity and internal bond) is the recipe with 20% hardwood fibers, 80% softwood fibers and ≤5% bark. Although the values of the surface absorption are the highest with this recipe, it can be also noticed that it is one of the few recipes for which the surface absorption on the back is lower than on the face (by 10.7%), which is an essential advantage.

Influence of the the Release Agent Upon the Physical and Mechanical Properties of HDF Panels

Spraying the panel surface before pressing is a common procedure to avoid the sticking of the HDF sheet on the press platen. The authors found it interesting to investigate how and to which extend the type of this release agent also influences the physical and mechanical properties of the panel. An improvement of the surface absorption, respectively a more pronounced reduction of the surface absorption on the panel back compared to the panel face was envisaged especially.

Considering the results obtained with different composition recipes, the one with 20% hardwood fibers, 80% softwood fibers and less than 5% bark was selected to be tested with three agents: water; the release agent type PAT 2003 XE (by Würtz) and the release agent type MOULEX WE07BSP (by Additek).

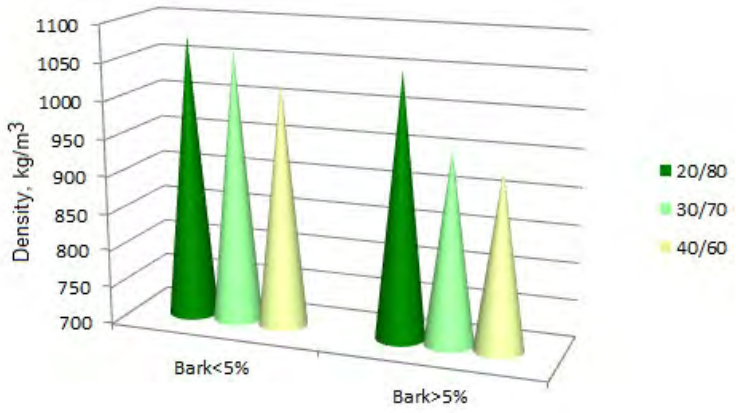
The results are presented in Table 3.

Table 3

Influence of the release agent upon the properties of 3mm thick HDF panels* – mean values and (standard deviations)

Release agent	Density, kg/m^3	Surface absorption on: face, mm	back, mm	Bending strength, N/mm^2	Modulus of elasticity, N/mm^2	Internal bond, N/mm^2
None	1078 (26.55)	446 (38.62)	398 (34.68)	79.60 (3.28)	6806 (196.84)	2.52 (1.31)
Water	1076 (22.39)	443 (21.93)	380 (14.00)	60.36 (5.30)	4493 (356.24)	1.5 (0.81)
PAT 2003 XE (by Würtz)	1026 (38.25)	397 (41.68)	350 (31.43)	74.09 (5.14)	6233 (446.35)	1.84 (0.96)
MOULEX WE07BSP (by Additek)	1005 (25.37)	333 (15.70)	246 (10.07)	71.36 (3.24)	5380 (369.76)	1.40 (0.78)

** values for composition recipe with 20% hardwood fibers, 80% softwood fibers and >5% bark.*



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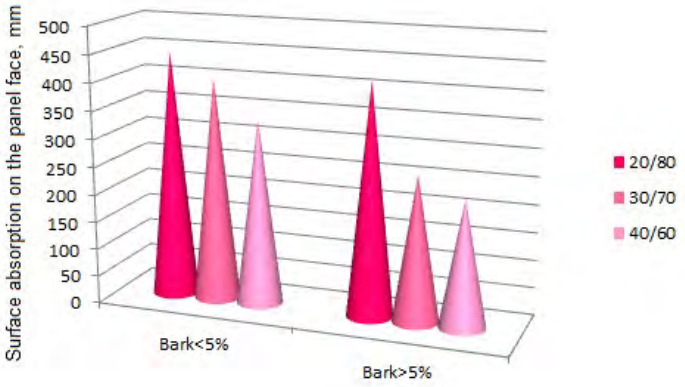

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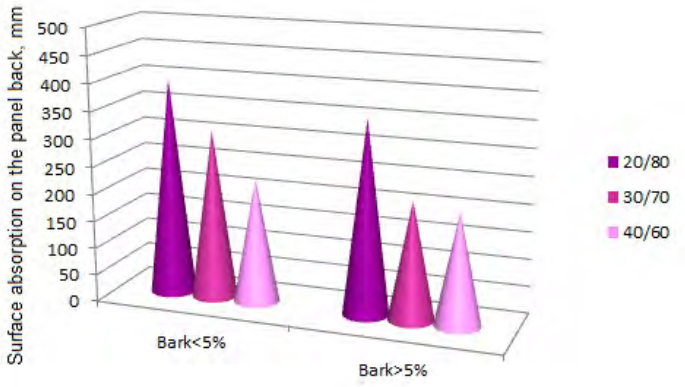

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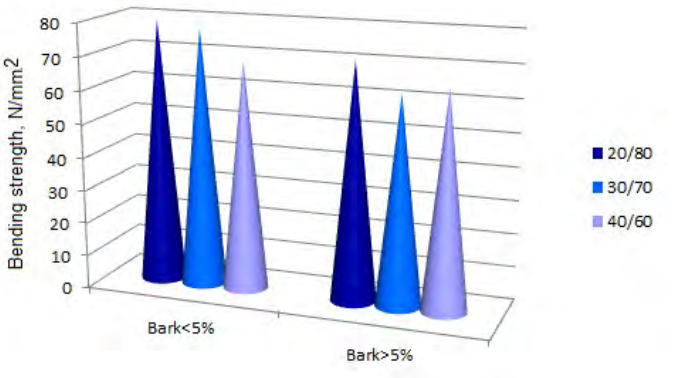

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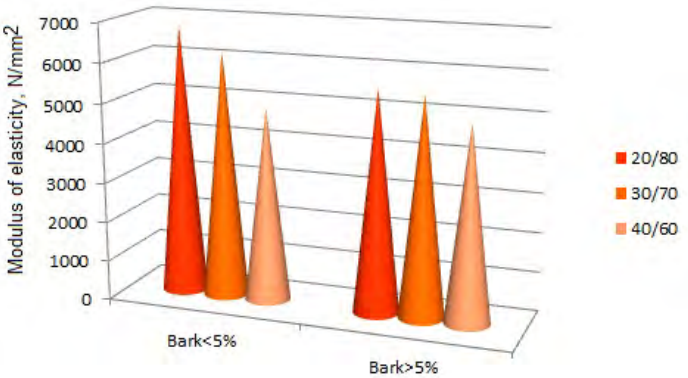

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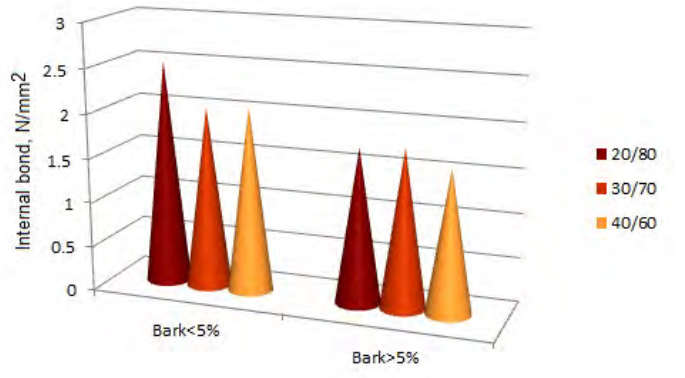

f.

Fig. 7. Influence of composition recipe upon physical and mechanical properties of HDF panels:
a – density;
b-surface absorption on the panel face;
c-surface absorption on the panel back;
d-bending strength;
e-modulus of elasticity in bending;
f-internal bond.

As one may notice, all mechanical properties are lowered by the use of the release agent. The lowest (worst) values were obtained when spraying with water (24.1% reduction of bending strength, 34% reduction of elasticity modulus and 40.5% reduction of internal bond) and the best when using the release agent PAT 2003 XE (by Würtz): only 6.9% reduction of bending strength and 8.4% reduction of elasticity modulus and 27% reduction of internal bond.

The density remained close to the one of the witness test pieces (with no spraying) only in the case of spraying with water. With the two release agents, it decreased by 4.8%, and by 6.7%, respectively.

As far as the surface absorption is concerned (Fig. 8), the most significant reduction was obtained with the MOULEX WE07BSP release agent: by 25.3% on the panel face and by 38.19% on the panel back. However, it must be mentioned that after a certain time of use, the release agent begins to foam, which is a disadvantage, because an anti-foaming additive has to be added.

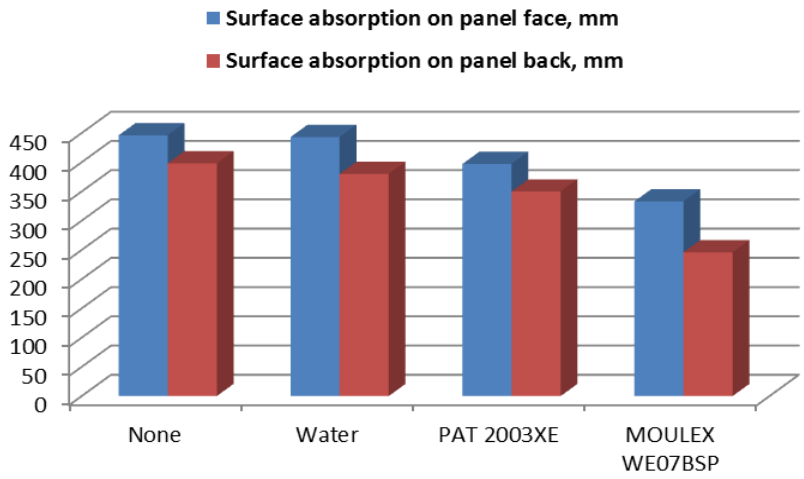

Fig. 8. Influence of the release agent upon the surface absorption of HDF panels.

The second best results concerning the reduction of the surface absorption were obtained with the Würtz PAT 2003 XE release agent: reduction by 11% on the panel face and by 12% on the panel back. However, the absence of foaming is a strong point in favor of this release agent.

The values presented in Table 3 enable each producer to choose according to his own priorities.

CONCLUSIONS

The conclusions of the present research can be formulated as follows:

1. By varying the raw material recipe from 20% hardwood fibers + 80% softwood fibers to 40% hardwood fibers + 60% softwood fibers, all physical and mechanical properties of 3mm thin HDF were lowered:
   - the density decreased by 4.9-10.9%;
   - the surface absorption decreased by 24.4-45.5%;
   - the bending stength decreased by 8.9-13.4%;
   - the modulus of elasticity in bending decreased by 11.7-28.1%;
   - the internal bond decreased by 8.0-11.7%.
2. The bark content in the composition recipe is also very important: with bark contents >5%, all HDF properties decrease. As an example, for the 20%/80% recipe with bark>5% compared to the one with bark ≤5%:
   - the density was by 2.6% lower;
   - the surface absorption on the panel face was by 5.8% lower;
   - the surface absorption on the panel back was by 10.8% lower;
   - the bending stength was by 10.5% lower;
   - the modulus of elasticity in bending was by 8.6% lower;
   - the internal bond was by 30.9% lower.
3. Thus, the composition recipe which was considered optimum, especially considering the mechancial performances was the one with 20% hardwood fibers, 80% softwood fibers and ≤5% bark, although it registered the highest values of the surface absorption.

4. The improvement (reduction) of the surface absorption is possible by using release agents, although this measure leads to some mechanical weakening. The best results (lowest values both on the face and on the back of the panel + surface absorption on the panel back distinctive lower than on the panel face) were obtained with the MOULEX WE07BSP release agent: 333mm surface absorption on panel face compared to 446mm (with no spraying) and 246mm surface absorption on panel back compared to 398mm (with no spraying). The PAT 2003 XE release agent by Würtz was remarked due to the absence of foaming and better mechanical performances, close to the ones obtained with no spraying.
5. The obtained results are applicable at any HDF producer, and can be used for process optimization.

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EVALUATION OF ORIENTED STRAND BOARD BEHAVIOR ON FIRE

Octavia ZELENIUC
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: zoctavia@unitbv.ro

Luminiţa BRENCI
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: brencilu@unitbv.rp

Cosmin SPIRCHEZ
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: cosmin.spirchez@unitbv.rp

Abstract

Wood based panels including oriented strandboard (OSB) are being used quite extensively on the construction market, therefore the fire performance knowledge is required. The aim of this paper was to evaluate the performance to fire of treated and untreated OSB panels compared to untreated and treated softwood panels. The tests were performed on the laboratory conditions based on the SR 652 method, analysing the weight loss of specimens subjected to combustion. Three type of fireproofing products (receipts: R1, R2 and R3) were applied by superficial treatments (brushing and dipping). The results indicated that the treated OSB and softwood had a good behavior to fire, at a retention level of about 280g/m^2 and by dipping treatment, registering a lowest weight loss with receipt R3, below 10%, the standard limit being 30%. Treated specimens were more fire resistant than untreated ones which weight loss ranged between 38%-68% (the greatest value was acquired for OSB). Overall OSB performance to fire was comparable with softwood but burning was occured more rapidly as can be seen in higher weight loss during test.

Key words: *weight loss; treatment; solid softwood; OSB; fire-retardant.*

INTRODUCTION

Oriented strand board (OSB) is an engineered wood structural panel that has rapidly conquered the construction market replacing partially the plywood panels. OSB panels gained popularity for various applications like: subfloor underlayment, roof sheathing, wall sheathing, I-joists products and other industrial applications such as furniture, packaging, vehicle and wagon interiors etc. (APA 2000). The OSB production continued to increase reaching in 2011, in North America 13.5 million m^3, 4.7 million m^3 in Canada and 4.5 million m^3 in Europe (Eastin et al. 2012). Therefore, from general use to structural applications, OSB panels found success dominating more than half of the structural panel market (Ainsworth 2007; RISI 2015). An important issue in building construction, besides mechanical properties, is the fire performance. This property can be significantly improved by using proper chemical treatments. The most common fire retardant chemicals used for wood based panels are boron compounds (borax and acid boric), inorganic salts, phosphoric acid, ammonium sulfate, zinc chloride (Candan 2012; FPL 1999). The chemicals for treatment are incorporated with wood particles just before, during, or after gluing and wax-blending processes, by impregnation or other means during the product manufacturing (White 2006) obtaining the fire-retardant-treated (FRT) OSB panels. The OSB panels treated by surface methods are not included in this category. Surface coatings (dip, spray, or brush), or direct placement in the product (such as borate rods), are all post-manufacture treatments (PMT), and the main issue with these treatments is the chemical gradients within the product (Kirkpatrick 2006). For the most treated wood and wood based products, preservatives are applied by using pressure. However, sometimes this is not possible, and special facilities are required to do this treatment. As a consequence the surface treatments are used to apply the preservative chemicals.

The surface burning behavior and resistance to flame penetration are some fire performance properties of OSB panels studied by White (2006), along with the cone calorimeter test to assess the combustibility. The results provided by APA (The Engineered Wood Association) showed that

untreated OSB panels have a FSI (flame spread rating or index) between 127 and 172 depending on the panel thickness (0 is for noncombustible materials). The flame spreading index (FSI) varies between 0 and 200 being classified into: Class I (or A) 0–25, Class II (or B) 26–75 and Class III (or C) 76–200) (ASTM E 84). The lowest FSI is permitted for areas where the fire hazard is most severe and the highest values in rooms of most occupancy except hospitals (APA 2017; White et al. 2010). Fire-retardant-treated wood (impregnated by a pressure process) must have a FSI of 25 or less in accordance with ASTM E 84 test, with no evidence of significant progressive combustion, when the test is continued for other additional 20 minutes. Also the flame front shall not progress more than 3.2 m beyond the centerline of the burner at any time during the test (White and Winandy 2006). In the public buildings such as cinemas, theatres, libraries, schools, offices, hotels foyers and hospitals, where is a higher risk of fire, the fire retardant panels should be used. The flame retardant products in a building help to suppress the spread of fire and permit a safe evacuation of the persons. Wood frame walls, floors and roofs using conventional wood framing can be designed to provide up to 2 hours resistance to fire (CWC 2000).

There are 7 classes according to fire reaction, defined by EN 13501-1 (Eurocode 5): A_1, A_2, B, C, D, E and F (A1 & A2 are incombustible). The wood based products (among which are OSB panels) are included in D and E classes as combustible materials (medium to highly contribution to fire). The OSB 3* panels, having $\rho \geq 600$ kg/m^3 and thickness ≤8mm, belong to E class and D class in case of thickness greater than 9mm, respectively. These panels are classified as CWFT (Classification Without Further Testing).

* OSB/3 –load-bearing boards for use in humid conditions (acc. to EN-300:2007).

This classification, according to CEN (European Committee for Standardization) allows to identify rapidly the performance of standardized wood based panels used in construction, in classes specified in EN 1350-1, excepting OSB 3 specified above.

OBJECTIVE

The main objective of this research was to evaluate the OSB behavior on fire by weight loss and to analyse the fireproofing efficacy of surface treatment. Three types of fire retardant chemicals were used on OSB and solid softwood samples and the comparative performance of these two building materials was also investigated.

MATERIAL, METHOD, EQUIPMENT

OSB panels and solid softwood (spruce - *Picea abies L.*) acquiered from a store building materials were used for testing. Specimens of 150mm x 70mm x 10mm and 15mm respectively were cut from the conditioned panels (Fig. 2). The average moisture content of untreated samples was 6.5% for 10mm thickness and 7.7% for 15mm thickness. The average volum mass was 440kg/m^3 and 620kg/m^3 for softwood and OSB respectively. The tests were made based on SR 652: 2009, determining the samples weight loss after a combustion process in laboratory conditions using the equipment presented in Fig. 1.

Three types of fire-retardant chemicals for surface coating were applied on samples, by brushing (three layers) and dipping (20 minutes).

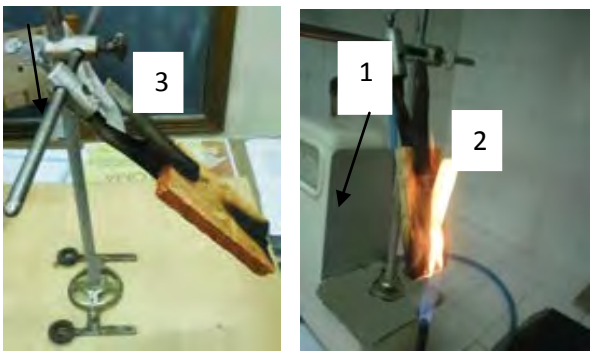
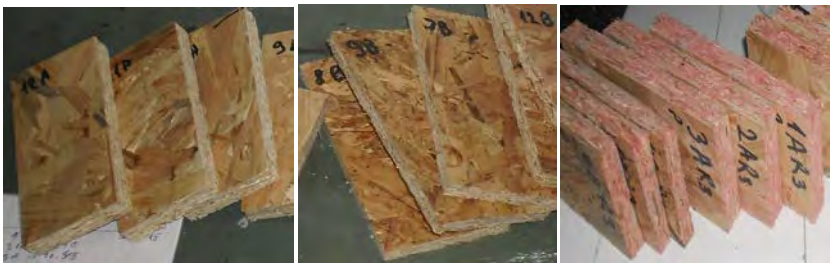


Fig.1
Device used in the fire experiments
1-scale, 2-sample, 3-device for clamping the specimen above the flame

The chemicals have been chosen based on literature studies (Ayrilmis 2006; Gao 2010; McIntyre 2004) and previous experience in the laboratory tests. The fire-retardant chemicals used

were a mixture of different chemicals as follow:

Receipt R1, boron compounds, phosphates, sulfates, isothiazolone, 25% concentration;
Receipt R2, amonium chloride and trisodium phosphate, 20% concentration;
Receipt R3, borax and zinc chloride, 20% concentration.



A.

B.

Fig. 2.
A. OSB treated samples: softwood treated samples

The samples were conditioned at 23±2ºC and relative humidity of 50±10% after treatment. Burning was performed in a room without air currents, at a temperature of (20±5)°C, using a gas burner flame with constant flow. The total duration of combustion process was set to10 minutes and at every 2 minutes was measured the weight loss. For each thickness, receipt and treatment, three specimens were tested. It was evaluated the fireproofing efficiency of treated and untreated samples.

Specific consumption and retention dose of fire-retardant chemicals

The fire-retardant coats the wood fibres providing a 'blanket' of protection, preventing the escape of flammable vapours and access of oxygen (Lowden 2013). Therefore, information on the retained dose of chemicals (d_r) (2) in panels it is important and it was calculated on the basis of specific consumption (C_{sp}) (1).

$$C_{sp} = \frac{M_{tf} - M_i}{A} \quad [g/m^2] \tag{1}$$

$$d_r = c \cdot C_{sp} \quad [g/m^2] \tag{2}$$

where:
$M_{i,}$ is the initial mass of sample after treatment, in g.
M_{tf}, is the final mass of sample, after treatment, in g.
c, is the concentration of fire retardant solution, in %.

Weight loss

The weight loss (WL), was calculated for each treatment and receipt according to formula (3):

$$WL = \frac{M_0 - M_f}{M_0} \cdot 100 \quad [\%] \tag{3}$$

where:
M_0 is the initial weigh of specimens, (before ignition), in g.
M_f is the final weigh of specimens, after combustion, in g.

Additionally, density (ρ) of each specimen was determined, knowing that besides thickness, chemical constituents and test procedure, density is another factor that influence the flame spread (White 2000) and charing process.

RESULTS AND DISCUSSION

Specific consumption and retention dose

The average specific consumption of OSB specimens ranged between 84-278g/m^2 with some differences between thicknesses (Table1). The lowest values were obtained for the samples with small thickness (10mm) treated by brushing, because of uneven distribution of chemicals on the specimens surfaces. In dipping treatment the specimens are completely submerged in the treatment solution thus a better penetration was achieved.

Table 1

The specific consumption for treated OSB samples

Fire retardant Receipt	Treatment applied/Thickness			
	Brushing		Dipping	
	10mm	15mm	10mm	15mm
	Average specific consumption, g/m^2			
R1	84	132	100	118
R2	104	123	142	201
R3	195	202	237	278

Table 2

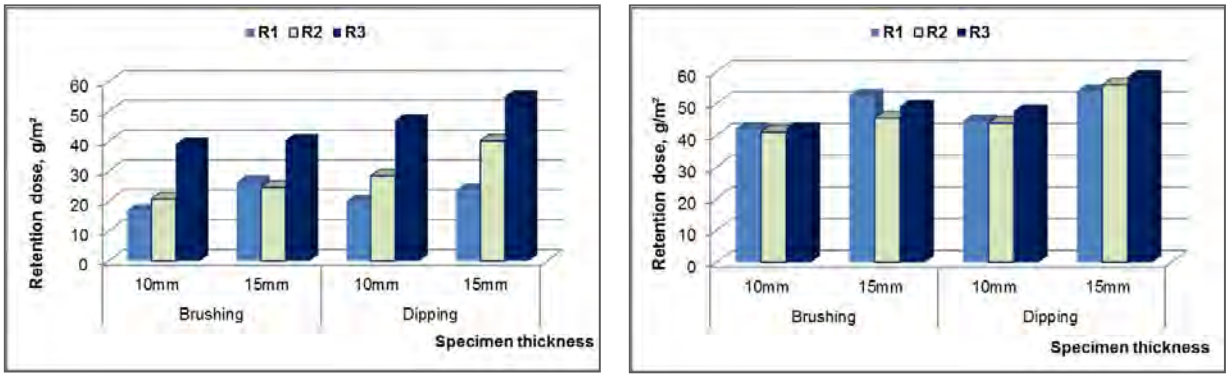
The specific consumption for treated softwood samples

Fire retardant Receipt	Treatment applied/Thickness			
	Brushing		Dipping	
	10mm	15mm	10mm	15mm
	Average specific consumption, g/m^2			
R1	168	210	163	216
R2	208	227	219	279
R3	212	245	238	292

In Table 2, the average specific consumption of fire-retardant chemicals in softwood samples is presented. The higher values resulted for softwood samples compared to OSB samples, obviously due to the anatomical structure of wood and its highly porous nature.

Retention values, as can be seen in Fig. 3 depended on the panels types (OSB and solid wood), thickness, treatment applied and receipts.

It is mentioned that the same concentration of chemicals level was used for the receipts R2 and R3 and the specimens had the same moisture content. The retention dose (d_r) for OSB, ranged between 17g/m^2 and 55g/m^2 and between 41g/m^2 and 58g/m^2, for softwood samples, with the greatest values for 15mm thickness and dipping treatment. Softwood specimens showed no significant differences between receipt and thickness when compared to OSB specimens.



A. **B.**

Fig. 3
Average retention dose for OSB (A) and softwood (B) treated samples.

The retention dose was with about 38% and 28% lower in case of brushing and dipping respectively, when compared OSB with softwood specimens, due to differences in porosity and surface wettability.

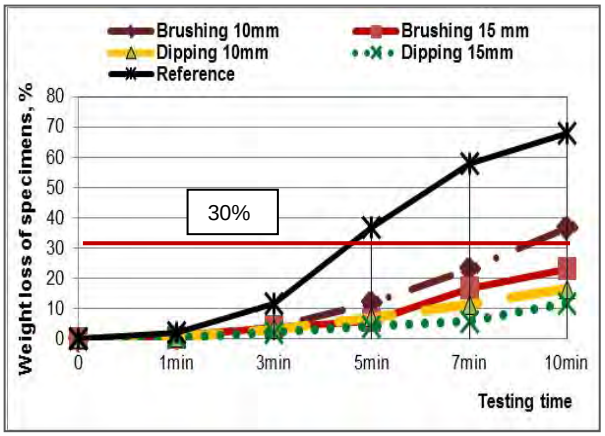
The greatest values of retention level was registered for both OSB and softwood specimens, in case of dipping treatment with the receipt 3 (R3). Receipt 3 included borax and zinc chloride. Due to their effectiveness as a preservative, and relatively low impact on the mechanical properties of wood, boron compounds are often preferable to other fire retardants (Anonymous 1999, Lebow and Winandy 1998). Zinc chloride was found also the most succesful retardant chemical in LVL (Laminated veneer lumber) (Kol 2010). Low perforance was achieved by brushing treatment, because of uneven application, some areas probably being not well penetrated by chemicals.

Weight loss

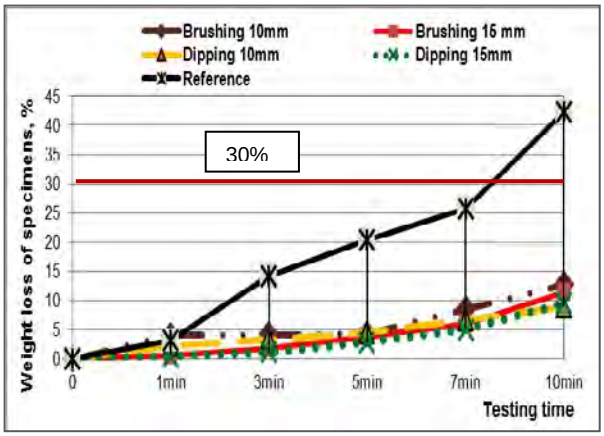
All chemically treated samples had lower weight loss values than those of untreated ones (reference samples) and almost all are below 30%. Untreated samples had the weight loss ranged between 58%-68% and 38%-42% for OSB and softwood respectively.

The evolution of weight losses during test is shown in Fig. 4 A-F, for each receipt and specimen thickness. Horrizontally line (30%) represents the maximum limit up to which the treatment is effective fireproofing according to SR 652. The burning process occured more quickly at OSB specimens,although the ignition time was higher (120 sec.) than for softwood specimens (60-80 sec.). This behavior could be determined by OSB mass volum, composition, however strands wood burned more easily than solid wood no matter the specimen thickness. After 2 minutes all OSB treated specimens start to loss in weight and after 5 minutes the weight loss was almost double.

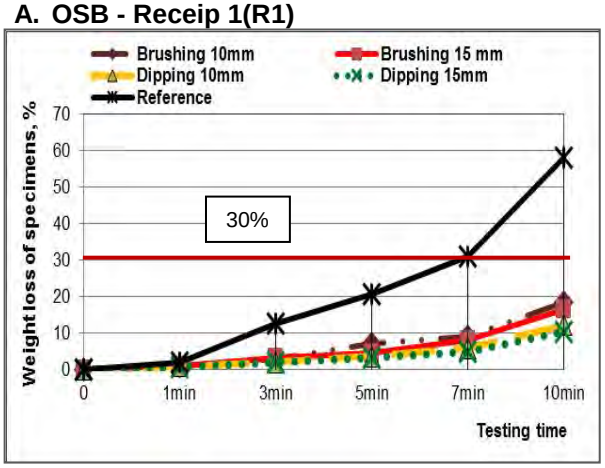
The greater weight loss was acquired in the brushing treatment for all receipts, the poor retention dose didn't succeed to provide sufficient protection to fire. Effectiveness of brushing with R1 was very low on OSB 10mm (Fig. 4A), which didn't pass the limit of 30% recommended by standard SR 652.



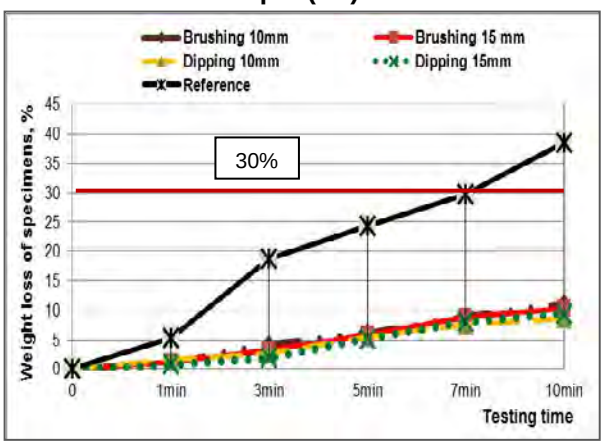
A. OSB - Receip 1(R1)



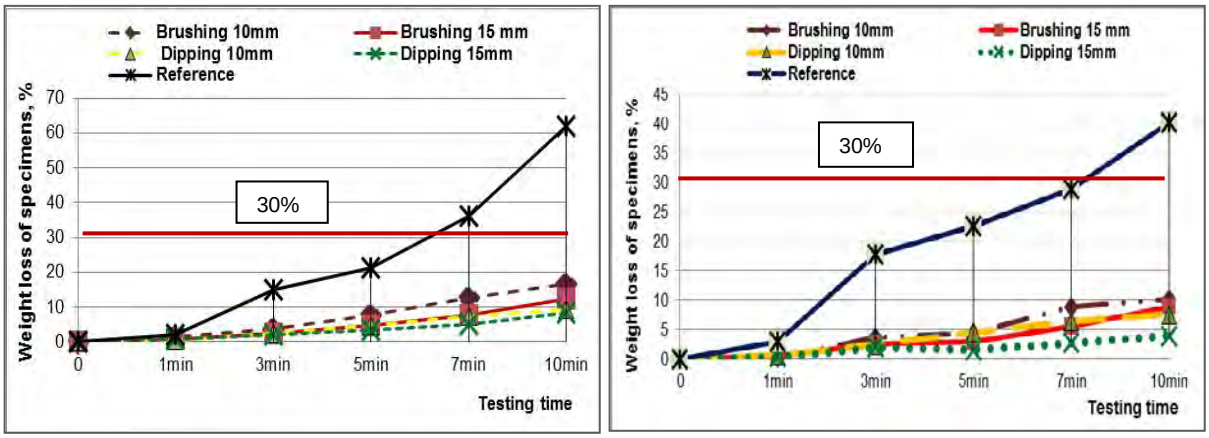
B. Softwood - Receipt 1(R1)



C. OSB - Receipt 2 (R2)



D. Softwood - Receipt 2 (R2)



E. OSB - Receipt 3 (R3) **F. Softwood - Receipt 3 (R3)**

Fig. 4
Fireproofing efficacy by weight loss, on OSB and softwood depending on receipt and thickness of the specimen

The lowest weight loss values registered during test were observed in the specimens treated by dipping with R3 (3.84 % and 8.25% for softwood and OSB respectively), followed by R2 (9.49% and 10.20%) and R1(9.52% and 11.66%). The weight losses registered for OSB were with about 25% greater in case of R1 compared to R2 and R3 treatments. Smaller differences (below 10%) was observed in softwood specimens treated with R1 compared to R2 and R3 respectively.

All specimens treated by dipping with R3 had the weight losses below 10%. Dipping treatment improved the fire performance of OSB, however the solid softwood has better behavior to fire regarding the weight loss after 10 min testing.

CONCLUSIONS

The results obtained in this study showed that treated samples had better fire performance than untreated ones. OSB samples trated by dipping with R3, had comparable performance with softwood, regarding the weight loss acquierd after 10 minutes fire test. The burn rate was higher in case of R2 and R1, for OSB greater weight loss values being recorded compared to softwood. The best fire performance had the samples treated with R 3, followed by R 2.

The weight loss performance of the panels was positively affected by the fire retardant chemicals, especially when applied by dipping when higher retention was acquiered by the thick specimens. Surface applications like brushing, is easy to perform but offers only a slight protection in terms of fire perormace compared to dipping treatment.

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EFFECT OF RAW MATERIAL COMPOSITION OF WOOD PLASTIC COMPOSITES ON SURFACE ROUGHNESS PARAMETERS EVALUATED WITH A ROBUST FILTERING METHOD

Lidia GURAU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: lidiagurau@unitbv.ro

Nadir AYRILMIS
Istanbul University, Forestry Faculty, Bahcekoy, 34473, Sariyer, Istanbul Turkey
Tel: 0090 212 338 2400 Fax: 0090 212 338 2424, E-mail: nadiray@istanbul.edu.tr

Abstract

This study extensively investigated the surface roughness of injection molded wood plastic composites (WPCs) produced from different amounts of wood flour, polymer matrix, mineral filler, and other additives. A larger range of roughness parameters than previously used in the literature were obtained from nine different WPC compositions by using a robust filtering method (Robust Gaussian Regression filter) to have a better understanding of the overlaying quality of the samples. Three melt flow indices of the polymer were tested (MFI 3.6; 12 and 25).and it was found that WPCs produced with the PP having a MFI value of 25 were the smoothest. It was noticed that not only the wood flour percentage matters, but the combination wood flour-mineral filler is also important. The WPC compositions with lower polymer amount in favour of more wood flour and mineral filler, led to rougher surfaces. A decrease of wood flour in favour of increasing the mineral filler participation percentage had a surface smoothing effect. At the same wood flour content, a decrease in polymer combined with an increase in mineral filler, led to rougher WPC surface. Among the tested WPC compositions, the smoothest corresponded to a participation of 50 wt% wood flour, 0 wt% mineral filler and around 40 wt% polymer. The results should be helpful to anticipate the effect on surface roughness of the percentage participation for each amount of the wood or mineral filler, polymer matrix, and additives in further development of WPC combinations.

Key words: *wood plastic composite; robust filtering method; surface roughness; filler; polypropylene.*

INTRODUCTION

Surface coatings and overlaying of wood-based composites such as particleboard and fiberboard are directly affected by surface quality. Surface roughness is one of the significant parameters affecting the surface quality wood based composites (Zhong and Hiziroglu). Wood plastic composites (WPCs) are gaining popularity in wood-based composite industry for nonstructural exterior applications including decking, fencing, siding and paneling. Hence, the evaluation of surface roughness of WPCs is needed. Finishing properties of WPCs are mainly dependent upon the properties of the raw materials such as polymer and wood flour content and characteristics, and manufacturing parameters. For the direct painting, the surfaces of WPCs have to be smooth, stable, and not highly absorbent. Therefore, it is important to quantify surface roughness of the panel to have a better overlaying of the WPC. Previous studies reported that surface roughness of WPCs was significantly affected by the type and ratio of the thermoplastic, wood, and additives (Jarusombuti and Ayrilmis 2011; Ozdemir and Mengeloglu 2008). However, evaluation of surface roughness in case of wood or of any wood based composite requires a special consideration. It was found that general standard requirements followed by the vast majority of researchers, more likely valid for homogeneous materials as metals, are not applicable for wood (Tan et al. 2012; Gurau et al. 2006; Krisch and Csiha 1999).

The inherent wood anatomy is a source of bias, from the selection of the measuring instrument to the evaluation of measured data (Fujiwara et al. 2004; Gurau 2004). The most important biasing effect of wood anatomy is caused during the filtering of the roughness (short wavelength irregularities) data from waviness (long wavelength irregularities). Most common filters such as the simple Gaussian filter from ISO 11562 (1996) + Cor 1 (1998), now replaced by ISO 16610-21 (2011) and ISO 13565-1 (1998) are causing a distortion known as "push-up" (Krisch and Csiha 1999, Gurau 2004), which makes the result of filtering unreliable. The artificial "push-up" occurs in case the surface

contains isolated deep valleys or high peaks, both type of irregularities being common when wood is used as such or in composites. However, a Robust Gaussian Regression Filter (ISO/TS 16610-31) was found robust and recommended for wood surfaces. The robust filter was tested on wood surfaces since it was a draft version and found useful (Fujiwara et al. 2004; Gurau 2004). More recently, these findings were confirmed by Tan et al. (2012) and Piratelli-Filho et al. (2012). If the RGRF works well on wood surfaces, can work reliably for any surface that contains wood ingredients (wood fibers, wood chips, wood flour), which all may be a source of bias and distortion during filtering with the common simple Gaussian filter. Furthermore, researchers generally used a limited number of roughness parameters, from the ones available in the standards, to evaluate the surface quality, such as *Ra* or *Rq* or a combination of *Ra* and *Rz*. A thorough understanding of the surface condition is needed, especially for composite materials where wood is used in combination with other materials and so introducing a degree of uneveness. Gurau et al. (2011) interpreted a range of profile roughness parameters from ISO 4287 (1997) and ISO 13565-2 (1998)] when applied to wood and found that apart from *Ra* or *Rz*, parameters as *Rq*, *Rt*, *Rsk* and *Rku* (from ISO 4287) as well as (*Rk*, *Rpk* and *Rvk* from ISO 13565-2) give refined information about the presence of deep valleys, accidental gaps in the surface and surface fuzziness outlying from a core roughness. The usefullness of some shape parameters as *Rsk* and *Rku* for evaluating the WPC surfaces was also acknowledged by Hutyrová et al. (2016).

OBJECTIVE

In this study, a large number of surface roughness parameters were used from roughness profiles obtained after robust filtering with RGRF of the injection molded WPCs produced from different amounts of the filler, polymer matrix, and additives, to have a better understanding of the overlaying quality of the samples.

MATERIAL, METHOD, EQUIPMENT

Virgin PP granulates were used as polymer matrix in the production of thermoplastic composites. The density and melt flow rate (230°C / 2.16kg) of the PP were 0.904g/cm^3 and 12g/10min, respectively. Pine wood (*Pinus sylvestris*) chips without bark were grinded in the grinder and then sieved to obtain wood flour having 0.25mm size. The wood flour was dried in a laboratory oven with a fan at 100°C for 24h to the moisture content of 0-0.5% before the manufacturing process. The particle size of the mineral filler (calcite) was 0.15mm. The amounts of compatibilizing agent, maleic anhydride modified homopolymer PP (MAPP), mineral filler, wax, UV absorber, zinc borate, antioxidant, and color pigment are given in Table 1.

Table 1

Experimental design

Type of raw material	Raw material composition of WPCs (wt%)								
	A1	A2	A3	A4	A5	A6	A7	A8	A9
Polypropylene	40.1 (MFI: 3.6)	40.1 (MFI: 12)	40.1 (MFI: 25)	28 (MFI: 12)	30 (MFI: 12)	29 (MFI: 12)	43.7 (MFI: 12)	39.7 (MFI: 12)	22.5 (MFI: 12)
Wood flour	50	50	50	60	40	50	50	50	60
Mineral filler	0	0	0	0	20	10	0	0	10
Coupling agent	3	3	3	3	1.5	2	3	3	3
Wax	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
UV-absorbent	0	0	0	0.5	0.5	0.5	0	0	0.5
Zinc borate	4	4	4	4	4	4	0	4	0
Antioxidan	0.4	0.4	0.4	0.7	0.8	0.8	0.8	0.8	0.8
Color pigment	0	0	0	1.3	0.7	1.2	0	0	0.7
Total amount	100	100	100	100	100	100	100	100	100

Preparation of Injection Molded WPC Composites

The oven-dried wood flour and mineral filler with a moisture content of less than 0.5wt%, PP granulates, and other additives were weighed for each formulation according to Table 1. All the materials were then dry blended in a drum type mixer for 5min before the extrusion. Following the pre mixture, all the materials were compounded with a co-rotating twin-screw extruder (L/D: 38) having a barrel temperature ranging from 175°C (feeding zone) to 185°C (the die zone), at a screw speed of 60 rpm for 14min. The compounded materials were cooled down by carrying them through water and pelletized. The pellets were then dried in an oven at 90°C for 24h before the injection molding process. Finally, the pellets were directly injection molded to produce test specimens using an injection molding

machine. The barrel temperature of the injection molding machine was controlled between 160°C and 190°C at 6MPa pressure. The specimens were conditioned at a temperature of 23°C and relative humidity of 50% until a constant mass was reached according to ISO 291 (2008). The density values of the composites ranged from 1.05 to 1.11g/cm^3. The experimental design is given in Table 1.

Method

A surface roughness methodology for measuring and evaluation of surface data proposed by Gurau et al. (2012) was used in this research. The methodology was born from previous research experience, which indicated a stylus instrument as reliable for wood surfaces. The measurements were performed on the WPC surfaces by using a MarSurf XT20 instrument manufactured by Mahr GmbH, Göttingen, Germany, endowed with a scanning head MFW 250 with tracing arm in the range of ±500μm and a stylus with 2μm tip radius and 90° tip angle, which measured the specimens at a speed of 0.5mm/s and at a low scanning force of 0.7mN. The instrument had MARWIN XR20 software installed for processing the measured data.

The surface roughness measuring lengths of WPC's were generally rather short: 4mm (Hutyrová et. al (2015)) or 12.5mm (Ayrilmis et al. 2012; Jarusombuti and Ayrilmis 2011; Ozdemir and Mengeloglu 2008). However, in this research, the specimens (four replicates for each WPC combination) were scanned on tracing lengths of 50 mm, because longer tracing length gives more accurate results (2012). Four profiles were recorded for each specimen, two on each specimen face, so that a total of 16 profiles were available for further evaluation of parameters for each WPC combination. The profiles were measured in areas which displayed, as much as possible, homogeneity of the mixture. The lateral measuring resolution was set for 1μm (50000 data points), so that any variation in surface irregularities could be detected with accuracy. The instrument provided a vertical resolution of 7nm.

The measured data was processed with the built-in software of the instrument. First, the software removed the form error and after that, the waviness. The roughness profiles were obtained by filtering each profile by using the robust filter RGRF (Robust Gaussian Regression Filter) contained in ISO 16610-31 (2010) and recommended in the methodology from Gurau et.al (2012). The cut-off used was 2.5 mm as recommended in previous research of Gurau et al (2006, 2012) and also used by various researchers specifically on WPC surfaces (Jarusombuti and Ayrilmis 2011; Ozdemir and Mengeloglu 2008).

A range of roughness parameters were calculated directly on the evaluation lengths, such as: *Ra*, *Rq* (they are mean parameters: the arithmetic mean of the absolute ordinate values, *Ra*, respectively, the root mean square value of the ordinate values, *Rq*), *Rt* (the total height of roughness profile calculated as the sum of the maximum profile peak height and the largest absolute value profile valley depth), *Rsk* (skewness calculated as the quotient of *Rq* cubed and the mean of the cubed ordinate values), *Rku* (kurtosis calculated as the quotient of Rq^4 and the mean of the ordinate values to the fourth power) from ISO 4287 (1997) and *Rk* (the core roughness), *Rpk* (the reduced peak height, which is the average height of the protruding peaks above the roughness core profile), *Rvk* (the reduced valley depth, which represents the average depth of the profile valleys projecting through the roughness core profile) from ISO 13565-2 (1998). The sum *Rk* + *Rpk* + *Rvk* was also determined for comparisons, because of parameters cumulative effect on surface roughness.

Mean parameters *Ra* and *Rq* are common roughness indicators, but alone, they do not provide sufficient information about wood surface topography. *Rt* is a parameter sensitive to the occurrence of high isolated peaks. *Rsk* and *Rku* are very sensitive to isolated extreme irregularities, which are not clearly detected by *Ra* or *Rq*. *Rsk* is a measure of the asymmetry of the amplitude density function and is a non-dimensional parameter. It is strongly influenced by isolated peaks or isolated valleys. Surfaces with a positive skewness, *Rsk* > 0, have fairly high peaks that protrude above a smoother plateau. Surfaces with a negative skewness, *Rsk* < 0, have fairly deep valleys in a smoother plateau. More random surfaces have skewness around zero indicating that the points are evenly distributed around the mean.

Rku is a measure of the sharpness of the amplitude density function and is a non-dimensional parameter. If is strongly influenced by isolated peaks or isolated valleys, which lead to a high kurtosis (*Rku* >3) in a data set; the probability density function tends to have a distinct peak around the mean, then declines rather rapidly.

Rk is a parameter which characterizes the depth of the roughness profile excluding protruding peaks and deep valleys. It corresponds to the highest concentration of datapoints and usually characterizes a processing process. *Rpk* and *Rvk* give information about datapoints located outside the core range and correspond to amplitudes defined outside the core by isolated peaks, respectively valleys. In combination with parameters from ISO 4287 (1997), these Abbot-curve parameters offer a

better understanding of the irregularities distribution along a measured profile and are a useful tool for comparisons between different surfaces. A mean value of roughness parameters and standard deviations were determined for each WPC combination.

Individual profiles of WPC combinations were also separately examined by using MathCad Professional from original measured data saved in ASCII. This allowed a visual comparison of the magnitude of irregularities between WPC compositions. The highest concentration of datapoints corresponding to the core data was separated with upper and lower thresholds from outlying peaks and valleys in the profiles with a method described in detail by Gurau et.al (2007). The core data should represent the effect of manufacturing the WPC composite as a mixture of materials and is characterized by the highest concentration of roughness datapoints above and below a reference line (zero line), which is generated by standard filtering. Datapoints above or below the core roughness delimited zone depict isolated features as peaks or valleys occurring less frequent on the surface in comparison with datapoints from the core. For example, the addition of wood, as heterogeneous material, to WPC can add isolated valleys extending below the core roughness attributed to the anatomical cavities. The adition of wood to WPC composites may be responsible also for isolated peaks caused by rised fibers.

RESULTS AND DISCUSSION

The results of the surface roughness measurements are presented in Table 2. WPC types A1, A2, and A3 had an identical composition, except for the MFI values. As for the melt flow index (MFI) of the PP, the lowest *Ra* value (1.5 μm) was found in the WPCs produced with the PP having a MFI value of 25. The WPCs produced with the PP having the MFI values of 3.6 and 12 had the same *Ra* value (1.6 μm). The fluidity of polymers increased with increasing MFI value. This resulted in better penetrating the wood cell cavities, which improved the surface smoothness of WPC measured not only by *Ra* and *Rq* parameters, but also by the core roughness parameter *Rk* and the composed parameter *Rk*+*Rpk*+*Rvk*, which gradually decreased from MFI 3.6 to MFI 25. An increased fluidity of the polymer in wood cavities is proved also by a gradual increase in negative *Rsk* in favour of valleys extending in isolation below the core roughness. Similarly, *Rku* increased as a result of more isolated valleys occurring while the polymer became more fluid. It can be remarked that roughness parameters for A1, A2, and A3 compositions in Table 2 were very similar and indicated the smoothest WPC surfaces together with A7 and A8 compositions. ANOVA test ($p<0.05$) confirmed that there were insignificant differences between the roughness parameters of these three groups (A1, A2 and A3). These groups of specimens had also the smallest *Rpk* values amongst all WPC combinations (Table 2. *Rpk* is responsible with the irregularities occurring above the core roughness, *Rk*. Furthermore, the standard deviation values for some of the most representative roughness parameters: *Ra*, *Rq*, *Rk*, *Rk*+*Rpk*+*Rpk* were the smallest amongst all WPC combinations (Table 2). From the compositions of A1, A2 and A3, A2 were compared with the other compositions, because the MFI of the polymer was the same, respectively MFI 12.

Table 2

Mean values roughness parameters (μm) for WPC composites made with different material ratios. Standard deviations are given in paranthesis

WPC type	Surface roughness and waviness parameters (μm)								
	Ra	*Rq*	*Rt*	*Rsk*	*Rku*	*Rk*	*Rpk*	*Rvk*	*Rk+Rpk+Rvk*
A1	1.6 (0.19)	2.4 (0.37)	27.0 (6.31)	0.2 (0.96)	8.6 (3.16)	4.4 (0.58)	3.6 (1.16)	3.0 (0.61)	11.0 (1.52)
A2	1.6 (0.18)	2.4 (0.37)	28.5 (6.71)	-0.2 (1.13)	10.4 (3.97)	4.3 (0.52)	3.6 (1.42)	3.6 (1.11)	11.4 (1.72)
A3	1.5 (0.15)	2.3 (0.30)	27.9 (5.45)	-0.8 (1.07)	11.3 (8.39)	4.0 (0.37)	3.0 (0.63)	3.6 (0.94)	10.7 (1.48)
A4	1.9 (0.36)	2.8 (0.65)	26.4 (6.44)	1.4 (0.84)	8.2 (2.64)	4.4 (0.81)	5.1 (1.85)	3.1 (1.03)	12.7 (2.66)
A5	1.7 (0.22)	2.6 (0.65)	32.5 (8.46)	1.1 (0.95)	10.1 (3.33)	4.0 (0.92)	4.9 (1.67)	3.1 (0.87)	12.0 (2.73)
A6	1.8 (0.40)	2.6 (0.68)	26.7 (9.99)	0.6 (1.00)	7.4 (2.16)	4.3 (0.85)	4.6 (1.72)	3.4 (1.17)	12.4 (3.15)
A7	1.6 (0.43)	2.5 (0.60)	29.0 (7.28)	0.9 (1.10)	9.6 (3.24)	3.8 (1.16)	4.5 (1.13)	3.2 (1.19)	11.5 (2.90)
A8	1.6 (0.40)	2.4 (0.61)	25.4 (5.71)	0.9 (1.05)	8.4 (1.91)	3.6 (0.97)	4.3 (1.43)	3.2 (0.93)	11.2 (2.70)
A9	2.2 (0.44)	3.2 (0.69)	35.5 (6.12)	0.2 (0.92)	8.2 (1.47)	5.2 (1.00)	5.2 (1.79)	4.5 (1.06)	15.0 (3.18)

The surface roughness of A2 was similar with the roughness of A7 and A8 compositions for the majority of parameters. It was noticed that those compositions, A2, A7, and A8 had the same percentage of wood flour (50%) and no mineral filler (Table 1). The compositions A2 and A8 differed slightly, by 0.4% less polymer in A8 compensated by the same more amount in additives (0.4% more antioxidant). Composition A7 had 3.6% more polymer than A2 compensated with 3.6% less additives (no Zinc borate, but 0.4% more antioxidant). These slight differences in composition did not change the surface roughness results as measured by *Ra*, *Rq*, *Rk*+*Rpk*+*Rvk*, but the core roughness *Rk* slightly decreased to 3.8 μm for A7 and 3.6 μm for A8 in comparison with 4.3 μm for A2, while peaks measured by *Rpk* increased to 4.5μm for A7 and 4.3 μm for A8 in comparison with 3.6 μm for A2 (Table 2, Figs. 1 and 2). The parameter *Rsk* was 0.9 for both, A7 and A8 compositions, in comparison with -0.2 for A2 (Tables 2 and 5). This shows again, a slight trend for the irregularities on the surface to display isolated peaks rather than valleys. It is not clear if these results can be attributed to the 0.4% addition of antioxidant in A7 and A8 compositions.

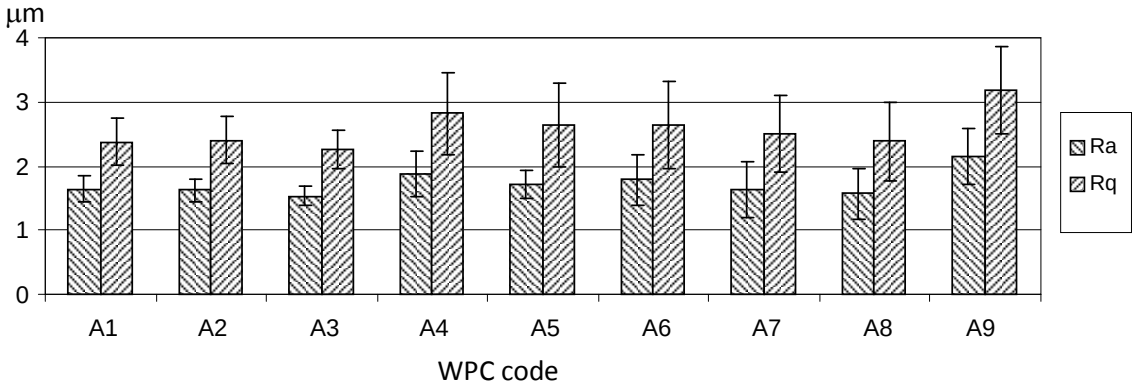

Fig. 1.
Variation of Ra and Rq roughness parameters (mean values and standard deviations) with the WPC composition.

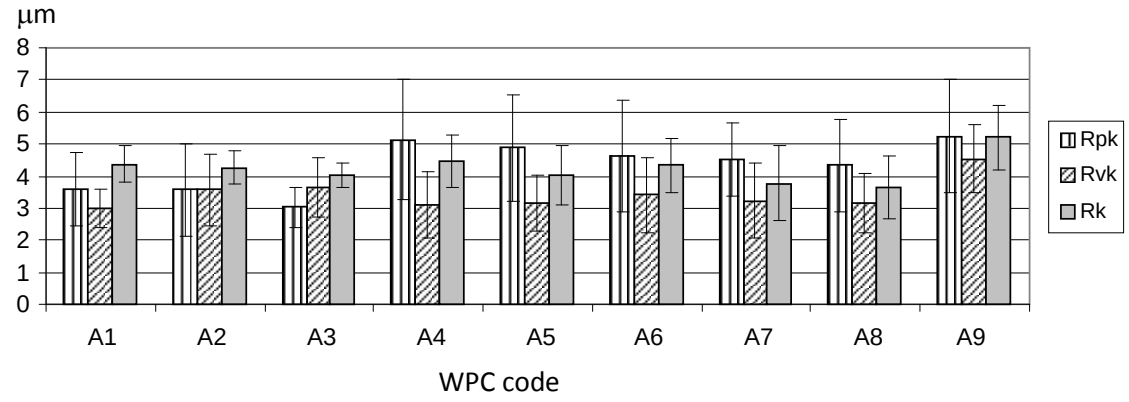

Fig. 2.
Variation of Rpk, Rvk and Rk roughness parameters (mean values and standard deviations) with the WPC composition

WPC surfaces having the highest wood flour percent (60%) had the highest roughness values (A9 followed by A4), but A5 having the smallest wood flour content (40%) wasn't the smoothest surface. It was noticed that not only the wood flour percentage matters, but the combination wood flour-mineral filler is also important. Thus, compositions having the highest wood flour- mineral content, contributed to the highest roughness values of WPC composites (Table 2). The roughest surface, judged by *Ra*, *Rq*, *Rk, Rpk, Rk*+*Rpk*+*Rvk*, was measured for A9, which had 70% wood flour - mineral filler (60% wood flour, 10% mineral filler). It was followed by A4, A6, and A5 with 60% wood flour-mineral filler (60% wood flour, 0% mineral filler in A4, 50% wood flour and 10 % mineral filler in A6 and 40% wood flour plus 20% mineral filler in A5). As the amount of mineral filler was replaced with wood flour in the composition, the surface roughness of the WPCs increased (Table 2). This was

attributed to the lower particle size (0.15 mm) of mineral filler as compared to the wood flour (0.25 mm). A4 and A9 had identical flour composition (60%), but A9 had 10% more mineral filler and 4.5% less additives. Reducing polymer content and additives in favour of mineral filler has increased the surface roughness of WPC to an *Ra* of 2.2 μm in A9 compared to 1.9 μm in A4 (Fig. 1). The core roughness *Rk* increased in A9 to 5.2 μm compared to 4.4 μm in A4. Similarly, *Rk*+*Rpk*+*Rvk* and *Rt* increased to 15 μm, respectively 35.5 μm in A9 in comparison with 12.7 μm respectively 26.4 μm in A4 (Table 2).

A6 and A5 have similar polymer content, 29% respectively 30%, but the A6 differentiated from A5 by the percentages of wood four and mineral filler. 10% more mineral filler complemented by 10% less wood flour in A5 led to a slightly smoother surface, which can be explained by the smaller size of mineral particles. Roughness parameters differed slightly: for example, *Ra* was 1.7 μm in A5 and 1.8 μm in A6, *Rk*+*Rpk*+*Rvk* was 12 μm in A5 and 12.4 μm in A6.

With regard to the polymer content, it can be seen from Table 1 and Table 2 that WPC compositions with the lowest polymer amount, A9 (22.5%), followed by A4 (28%), A6(29%), and A5 (30%) in favour of more wood flour and mineral filler, led to rougher surfaces in comparison with A7, for example, having the highest polymer percentage (43.7%). These differences were significant when analysed with ANOVA ($p<0.05$). The thresholds, represented with thick horizontal lines, separate the core roughness in the middle, which is higher in magnitude for A9 in comparison with A2. Also, by looking at the magnitude of peaks and valleys in those profiles, it is obvious they are higher in A9. This can be explained by a higher wood flour content in A9 (60%) than in A2 (50%) which added more fuzziness and wood anatomical valleys. 10% mineral filler in A9 has increased the core roughness *Rk* to 5.2 μm and the parameter *Rk*+*Rpk*+*Rvk* to 15 μm in comparison with A2, with 0% mineral filler and *Rk* of 4.3μm and *Rk*+*Rpk*+*Rvk* of 11.4μm. In general, the pretty high standard deviation values of the roughness parameters is an indication that surfaces are uneven and may require an improved mixing.

CONCLUSIONS

The surface roughness of various WPC compositions was evaluated by using a robust filtering method and a larger range of roughness parameters than previously used in the literature. The results showed that WPC goups A1, A2 and A3 were the most homogeneous and together with A7 and A8 provided the smoothest surfaces. The smoothest WPC compositions corresponded to a participation of 50% wood flour, 0% mineral filler and around 40% polymer. By increasing the fluidity of polymers, respectively the MFI value from 3.6, to 12 and 25, the surface roughness decreased due to a better polymer penetration inside the wood cell cavities. At the extreme, the roughest surface was measured for A9 (60% wood flour plus 10% mineral filler), followed by A4, A6, and A5, where the wood flour percentage alone or in combination with mineral filler was the highest. The WPC compositions with lower polymer amount in favour of more wood flour and mineral filler, led to rougher surfaces. It was noticed that not only the wood flour percentage matters, but the combination wood flour-mineral filler is also important. As the amount of mineral filler was replaced with wood flour in the composition, the surface roughness of the WPCs increased. Reducing polymer content in favour of mineral filler, while keeping the same wood flour percentage has increased the surface roughness of WPC. For similar polymer and additives participation, more mineral filler complemented by less wood flour led to a slightly smoother surface. These results should be helpful to anticipate the effect on surface roughness of the percentage participation for each amount of the wood or mineral filler, polymer matrix, and additives in further development of WPC combinations.

ACKNOWLEDGMENT

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STUDY OF THE SYNTHESIS PARAMETERS OF AN UREA-FORMALDEHYDE RESIN AND THEIR IMPACT ON PARTICLEBOARD PROPERTIES

Carolina GONÇALVES
LEPABE-Faculdade de Engenharia, Universidade do Porto
Rua Dr. Roberto Frias s/n 4200-465, Porto, Portugal
E-mail: carolina.goncalves@sonaearauco.com

João PEREIRA
LEPABE-Faculdade de Engenharia, Universidade do Porto
Rua Dr. Roberto Frias s/n 4200-465, Porto, Portugal
ARCP-Associação Rede de Competência em Polímeros
Rua Dr. Júlio de Matos 828/882, Porto, Portugal
E-mail: jamp@fe.up.pt

Nádia PAIVA
EuroResinas – Indústrias Químicas SA
7520-195 Sines, Portugal
E-mail: nadia.paiva@sonaearauco.com

João FERRA
EuroResinas – Indústrias Químicas SA
7520-195 Sines, Portugal
E-mail: joao.ferra@sonaearauco.com

Jorge MARTINS
LEPABE-Faculdade de Engenharia, Universidade do Porto
Rua Dr. Roberto Frias s/n 4200-465, Porto, Portugal
DEMad – Departamento da Engenharia de Madeiras, Campus Politécnico de Repeses
3504-510 Viseu, Portugal
E-mail: jmmartins@estgv.ipv.pt

Fernão MAGALHÃES
LEPABE-Faculdade de Engenharia, Universidade do Porto
Rua Dr. Roberto Frias s/n 4200-465, Porto, Portugal
E-mail: fdmagalh@fe.up.pt

Ana BARROS-TIMMONS
CICECO – Aveiro Institute of Materials and Departamento de Química, Universidade de Aveiro
3810-193 Aveiro, Portugal
E-mail: anabarros@ua.pt

Luisa CARVALHO
LEPABE-Faculdade de Engenharia, Universidade do Porto
Rua Dr. Roberto Frias s/n 4200-465, Porto, Portugal
DEMad – Departamento da Engenharia de Madeiras, Campus Politécnico de Repeses
3504-510 Viseu, Portugal
E-mail: lhora@fe.up.pt

Abstract

This paper presents the results for the optimisation of different synthesis variables. An industrial UF resin was synthesised using the alkaline-acid process (alkaline methylolation, acidic condensation and neutralization and finally the last urea addition) at different values of pH, temperature and final viscosity and characterised according to different analysis methods. The particleboards were also produced and characterised according to the standard tests. For this study, a statistical analysis using JMP software was performed, and the main conclusion is that small changes in the synthesis of resins variables do not affect the final performance of particleboards.

Key words: *urea-formaldehyde resin; particleboards; synthesis parameters; JMP Statistical Software.*

INTRODUCTION

In the last decades, the industry of wood products is going through a great evolution thanks to companies like Sonae Arauco, which focus has been on developing more and better wood-based products. In 2015, Portugal produced 1 million and three hundred thousand m^3 and exported 278 million euros of wood-based panels (FAO 2015). Among these products, the best known are the commercially available particleboard (PB), medium density fibreboard (MDF), oriented strand board (OSB) and plywood (PW). For all these types of panels the use of a synthetic adhesive is required. Among the wide range of adhesives/resins employed in the wood industry, the most important are the amino resins which include urea-formaldehyde (UF) resins, melamine-formaldehyde (MF) resins and melamine-urea-formaldehyde (MUF) resins.

Amino resins are thermosetting polymers and they are normally used in the production of wood-based panels, linings and high and low pressure laminates. There are essentially three types of these resins: urea-formaldehyde, melamine-formaldehyde and melamine-urea-formaldehyde. UF resins are commonly used in the manufacture of wood products, especially particleboard and medium density fibreboard, due to their high reactivity, low cost and excellent adhesion to wood (Dunky 2001). The major disadvantages are the low moisture resistance, and formaldehyde emission during the production and lifetime of the panels. Although the free formaldehyde levels of these resins have been declining over the past decades, the re-classification of formaldehyde by International Agency for Research on Cancer (IARC) as "carcinogenic to humans" in 2004, and the consequent emergence of more restrictive legislation, forced resin producers to develop a new generation of resins that lead to a decrease in formaldehyde emissions to the levels of natural wood (Carvalho *et al.* 2012).

The industrial production of UF uses the alkaline-acid process. This process is performed in three steps: alkaline methylolation, followed by an acidic condensation and neutralization and finally the last urea addition (Pizzi 2003). There is also an alternative process, the strongly acidic process in which the condensation step is carried out under strongly acidic conditions and occurs simultaneously with the methylolation step. This process leads to panels with low formaldehyde emissions without modifying any physical or mechanical properties, but it requires strict control of reagents supply and a high capacity cooling system (Ferra *et al.* 2011).

The most important factors that influence the resins properties are the formaldehyde/urea (F/U) molar ratio, the temperature and reaction time, and pH during the condensation step. Many studies have been carried out and different kinetic models proposed (Carvalho *et al.* 2006). However, the reversibility and the occurrence of intramolecular reactions leads to the formation of a great variety of chemical structures as methylene bridges, methylene ether, methylols, and even cyclic amide derivative groups, which makes the prediction of the properties of these resins a complex task (Costa *et al.* 2013).

The impact of the formulation of these resins in the performance of wood products was the subject of several studies, some of which used statistical tools as methodology to optimize the resins synthesis parameters in order to produce panels with maximum internal resistance and minimum formaldehyde emissions (Ferra *et al.* 2010; Guo *et al.* 2013). Some strategies to reduce formaldehyde emissions have been done directly in the resins: reducing the molar ratio Formaldehyde/Urea, doping with melamine and the addition of formaldehyde scavengers (Costa *et al.* 2013; Paiva *et al.* 2012). However, the strategies cause a loss of reactivity and cure rate, since formaldehyde is required for curing the resin. Thus, the suitability of the characteristics of UF and MUF resins for wood-based panels manufacturing is important to reduce formaldehyde emissions without changing physical and mechanical properties and without losing productivity. So, an optimisation of both resins synthesis and the production of wood-based panels (namely the pressing operation) becomes a crucial task.

OBJECTIVE

The main objective of the present research was to optimise different variables related to resins synthesis, trying to better understand their impact on wood-based panels properties, in particular particleboards. In an initial approach, an industrial UF resin was synthesised at different values of pH, temperature, and final viscosity. The resins were characterised using empirical quality control methods and advanced physicochemical characterisation techniques. The panels produced were characterised using standard tests. The results were then analysed using the JMP Statistical Software. With this study it will be possible to better understand the main variables of the process and how to change them.

MATERIAL, METHOD, EQUIPMENT

Formaldehyde (55 wt.% solution), urea, melamine, ammonium sulphate, sodium hydroxide (50 wt.% solution) and acetic acid (25 wt.% solution) were provided by Euroresinas – Indústrias Químicas,

S.A. (Sines – Portugal). Wood particles and paraffin for the production of particleboards were supplied by Sonae Arauco (Oliveira do Hospital – Portugal).

Resins production and characterisation

The resins were synthesised in a laboratory reactor. The synthesis was carried out in 2.5 L round bottom reactor, equipped with mechanical stirring and thermometer. A heating mantle heated the reactor and the temperature was controlled with a thermometer. The pH and viscosity measurements were performed offline on samples taken from the reaction mixture (and re-added after). All resins were produced according to the alkaline-acid process. These resins were divided in three series. Resins in the first series (resin A, B, C, D, E, F and G) were produced under different pH. The resins in second series (resin H, I, J and K) were produced under different temperature. Finally, the resins in third series (resins L, M and N) were produced with a different stop viscosity, between 250 and 600 mPa.s.

Common characterisation methods involved the determination of physical and chemical properties that are related to the resin performance, such as viscosity, solid content, gel time and pH. However, advanced methods, such as chromatography and spectroscopy techniques have been carried out, in order to provide more specific and detailed information of the structure and subsequent performance of the resins.

The resin pH was measured using a combined glass electrode. pH values for UF resins are usually between 7.5 and 9.0. The viscosity (mPa.s) value gives a rough indication of the degree of polymerization of the resin. Viscosity was measured using a Brookfield and/or Ford cup viscometer (ASTM 1200) at a constant temperature of 25 ºC. The resin density ($kg.m^{-3}$) is usually determined based on the weight/volume ratio and it can be measured using a hydrometer. The solid content (%) is determined by evaporation of volatiles in two grams of resin up to weight constant. Generally, this corresponds to three hours at 120 ºC. Gel time (s) is the time needed for the resin gelification under similar conditions of the hot-pressing process (at 100 ºC), after addition of a latent hardener. For this measurement, 100 g of a sample (diluted to 50 % solid content) was weighed in a beaker with 3 mL of a 30 % latent hardener. In a test tube 0.250 mL of the previous solution was added and it was immersed in boiling water. A rod was used for stirring the solution until resin gelification. HPLC is a chromatographic technique that allows separation of a mixture of different molecular weight compounds. This method is very effective in identifying low molecular weights (Ferra *et al.* 2010; Kumlin and Simonson 1978; Ludlam *et al.* 1986). The use of this technique in the analysis of UF resins allows the separation and identification of unreacted urea (% U), monomethylolurea (% MMU) and dimethylolurea (% DMU). A HPLC JASCO system equipped with a refractive index detector, JASCO IR-2031 Plus was used. The high-pressure pump used was a JASCO PU-2080 Plus pump. The column used was an YMC Polyamine II, conditioned at 30 ºC using an external oven JASCO PU-2067 Plus. The flow rate was 1.5 $mL.min^{-1}$ and acetonitrile/water (ACN/H_2O) was used as the mobile phase. The samples were prepared by dissolving 75 to 80 mg of resin in 1 mL of DMF, and after stirring for 1 minute, the mixture was diluted in 2 mL of 90 % of ACN and 10 % H_2O. When the mobile phase was added, flocculation occurred. The sample was then left to rest (10 minutes), filtered and then injected. The calibration was performed using urea and dimethylurea standards.

Particleboards production and characterisation

Wood particles were blended with resins, paraffin and catalyst in a laboratory glue bender. Surface and core layers were blended separately. The amount of resin in both surface and core layers was 7 wt.% (solid resin per dry wood particles).The catalyst amount in the core layer was 3 wt.% (dry catalyst per solid resin). Three layers particleboards were hand formed in a square aluminium deformable container with 220 x 220 x 80 cubic millimetres. Surface and core layer differ in particle size distribution and moisture content. The upper surface layer had a mass of 20 %, the core layer 62 % and the bottom surface layer 18 %. The pressing schedule of a continuous press is transposed to a batch cycle in a computer controlled laboratory press equipped with a linear variable displacement transducer (LVDT), a pressure transducer and thermocouples. For all series, eight boards were produced using four different pressing times (120, 150, 180, 210 s).

The boards were tested according to the European standards for density (D) (EN 323), internal bond (IB) (EN 319), moisture content (MC) (EN 322) and thickness swelling (TS) (EN 317).

Statistical analysis

Trying to better understand the resins synthesis process and their impact on wood-based panels properties, different variables related to resins synthesis were studied (Table 1). For confidentiality reasons the process variables temperature and pH are encoded. In a first approach, an industrial UF resin was synthesized at different values of pH, temperature, and final viscosity. These

parameters were analysed because they are crucial to the synthesis process. The values for this study were defined according to the medium value of the intervals of the variables. For UF synthesis, the process interval for pH of the first methylolation is between 8.0-10.0. As regards to the pH of condensation, the interval is 5.5-6.5. Finally, the pH of the second methylolation values are between 7.0-9.0. Thus, for UF synthesis, the process interval for pH of the first methylolation is $pH1_{MI}$-$pH3_{MI}$, the pH of condensation interval is $pH1_C$-$pH3_C$ and the pH of the second methylolation values are between $pH1_{MII}$-$pH3_{MII}$.

Also for temperature, three different values were analysed. For methylolation temperature, the values corresponding to $T1_M$, $T2_M$ and $T3_M$ ºC were studied; and for condensation temperature: $T1_C$, $T2_C$ and $T3_C$ ºC. The viscosity was the last parameter studied, and the goal of this study was to have resins with different final viscosities: 100, 150, 200 and 250 mPa.s.

The results were analysed using JMP Statistical Software after the characterisation of resins and particleboards. The main goal of the statistical analysis is to improve the internal bond and decrease the thickness swelling of particleboards.

Table 1

Factors and levels for statistical analysis

Factors	Units	Levels			
Methylolation I pH	-	$pH1_{MI}$	$pH2_{MI}$	$pH3_{MI}$	-
Condensation pH	-	$pH1_C$	$pH2_C$	$pH3_C$	-
Methylolation II pH	-	$pH1_{MII}$	$pH2_{MII}$	$pH3_{MII}$	-
Methylolation Temperature	ºC	$T1_M$	$T2_M$	$T3_M$	-
Condensation Temperature	ºC	$T1_C$	$T2_C$	$T3_C$	-
Final Viscosity	mPa.s	100	150	200	250
Pressing time	s	120	150	180	210

The parameters analysed in JMP can be divided in:

- Panel quality measurement (IB, TS);
- Resin properties (solids content (%), % U, % DMU, % MMU);
- Resins quality measurement (final viscosity, final pH, viscosity, pH, gel time, stability);
- Reaction parameters (pH methylolation/condensation, T methylolation/condensation, stop viscosity).

RESULTS AND DISCUSSION

In Table 2, the results for the resins characterisation for all produced resins are presented. An industrial resin is also presented for comparison. Related to viscosity and pH, all resins have the desirable value. The gel time is between 57-84 s and the solids content is between the defined values. All the resins had a normal stability (30 days). Comparing these resins (lab resins) with an industrial one (Ind) it is possible to notice that their characteristics are really similar.

Table 2

Characterisation of resins

Resin	Final viscosity (mPa.s)	Final pH	Viscosity (mPa.s)	pH	Gel time (s)	Solids content (%)
A	140	9.07	150	8.61	77	64.42
B	250	8.97	210	8.38	64	64.72
C	150	9.06	155	8.60	64	63.45
D	205	8.99	195	8.66	72	64.12
E	225	8.92	245	8.85	84	65.47
F	170	8.99	185	8.53	77	64.64
G	155	9.48	145	8.84	71	64.56
H	120	9.70	145	8.98	62	64.24
I	200	9.01	170	8.43	78	68.07
J	180	9.49	165	8.69	63	65.27
K	130	9.03	110	8.46	70	64.83
L	240	9.09	260	8.53	76	66.75
M	170	9.21	170	8.37	58	64.32
N	110	9.03	115	8.35	62	64.12
Ind	-	-	180	8.13	57	63.03

The first JMP analysis permitted to establish a relationship between panel quality and resin properties (Fig. 1). From the values presented in Table 3, it can be concluded that the % DMU significantly influences the IB, not being significantly influenced by other variables. This means that the IB is significantly affected by the % DMU but other variables hardly have any effect. The % MMU and % DMU significantly influence TS value. As the goal is to maximize the IB, this property was chosen as a control measure. Therefore, according to these results, it is desirable to have the highest % DMU, since the IB increases as the % DMU increases. These results can be explained with a widespread theory that a good UF resin should incorporate low molecular weight species that are important for penetration into wood, and higher molecular weight species that contribute to the cohesion of the particles. Thus, polymers with higher molecular weight should lead to an increase in the internal bond of the panels.

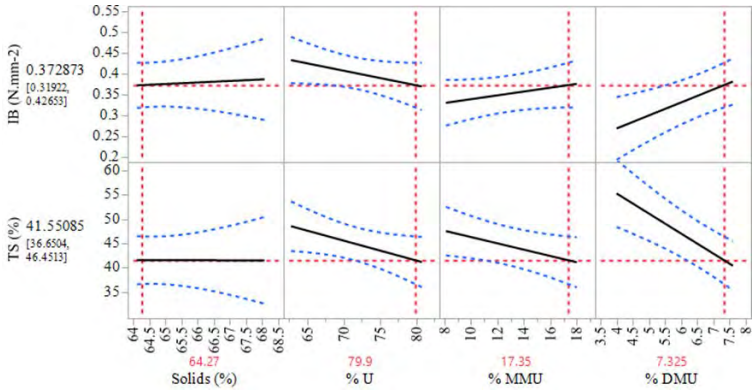

Fig. 1.

Effect of resin properties on panel quality (internal bond and thickness swelling).

Table 3

Parameters estimated and significance level (*5%, **1%, ***0%) for panel quality measurement as a function of resin properties

Factors	IB ($N.mm^{-2}$)	TS (%)
Solid content (%)	0.7544	0.9932
% U	0.1350	0.0580
% MMU	0.1984	0.0495*
% DMU	0.0046**	<0.0001***

The gel time is significantly affected by solids content and % U. By analysing the results, it is possible to conclude that a higher solids content corresponds to a less reactive resin. Regarding the solids content, it would make more sense for the gel time to decrease with increasing solids content. This is because that by decreasing the solids content, the concentration of the reactants decreases. Therefore, there is more water entering in the system which acts as a retardant to the curing of the resin. Thus, the cure rate decreases, decreasing the gel time.

The final viscosity and the next day viscosity are influenced by solids content and % U. The higher the solids the higher the viscosity, and the higher the % U the lower the viscosity. For the same amount of solids, the viscosity increases with an increase in the proportion of the condensate structures. As well, the proportion of molecules with high molecular weights increases with increasing the degree of condensation.

Stability as related to viscosity will depend significantly on solids content and % U. Stability decreases with the increase in solids content and increases with % U. With this analysis, it seems there is a relationship between the lower molecular weight particles and the stability of the resin.

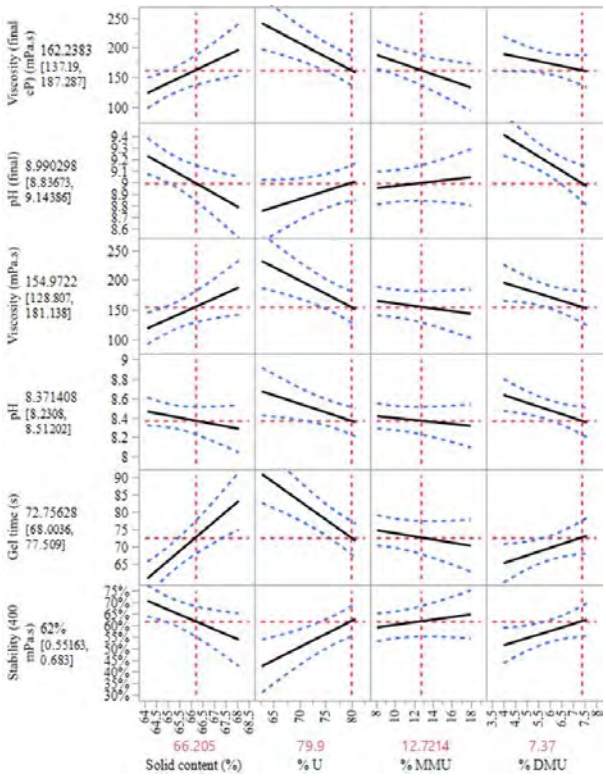

Fig. 2.

Resins quality measurement as a function of resin properties.

Table 4

Parameters estimated and significance level (*5%, **1%, ***0%) for resins quality measurement in function of resins properties

Factors	Final viscosity (mPa.s)	Final pH	Viscosity (mPa.s)	pH	Gel time (s)	Stability
Solid content (%)	0.0071**	0.0073**	0.0144*	0.2304	<0.0001***	0.0170*
% U	0.0009***	0.0843	0.0018**	0.0209*	<0.0001***	0.0018**
% MMU	0.0080**	0.4392	0.3068	0.3702	0.241	0.2862
% DMU	0.1818	0.0014**	0.0592	0.0241*	0.0580	0.0542

From the previous analysis and relating the IB to the % DMU, it was concluded that a higher % DMU in the resin yielded an increase of the IB in the final panel. Thus, analysing the reaction parameters with the properties of the resins and maximizing the value of % DMU, the "optimal solution" can be obtained for this set of results and for the resins under study. Through the statistical analysis performed, it asserted that the % DMU is significantly influenced by the stopping viscosity. This factor may be related to the fact that the stop viscosity is related to the condensation step and that a more condensed polymer will have a larger number of species with higher molecular weight. The solids content and % MMU are influenced by several factors and will not be considered in this analysis. The % U is influenced by T methylolation. This value increases with increasing T methylolation.

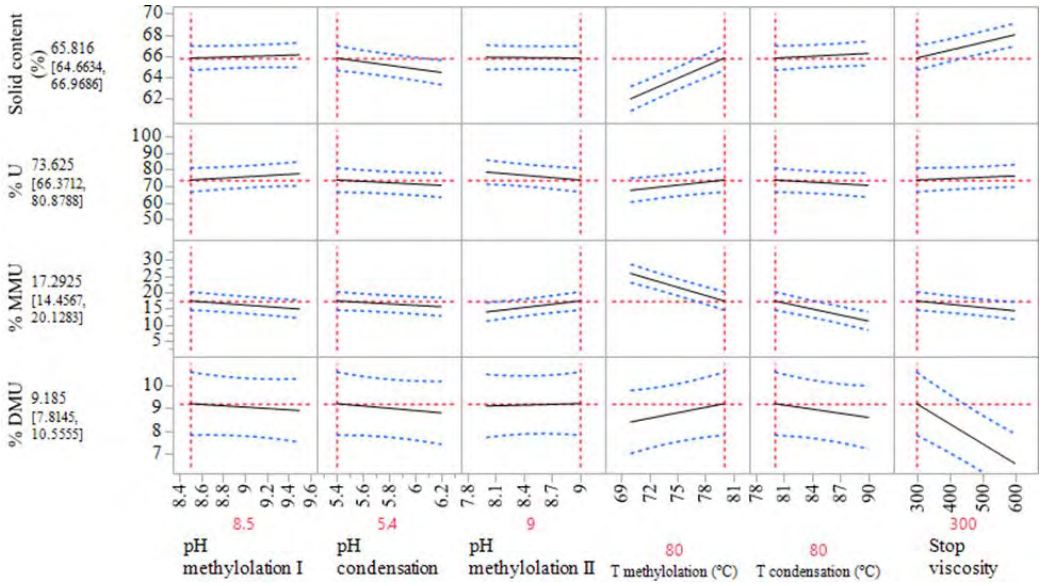

Fig. 3.

Reaction parameters as a function of resin properties.

Table 5

Parameters estimated and significance level (*5%, **1%, ***0%) for reaction parameters in function of resins properties

Factors	Solids content (%)	% U	% MMU	% DMU
pH methylolation I	0.5129	0.1909	0.0302*	0.5819
pH condensation	0.0047**	0.2695	0.1145	0.4634
pH methylolation II	0.8612	0.1002	0.0038**	0.8542
T methylolation	<0.0001***	0.0354*	<0.0001***	0.1458
T condensation	0.3385	0.2695	<0.0001***	0.2731
Stop viscosity	<0.0001***	0.3351	0.0038**	<0.0001***

After this study, the "optimal" resin was synthesised in laboratory using the correspondent factors levels, namely temperatures and pH for the methylolation and condensation and also stop viscosity that gave the best resin. The results obtained have demonstrated that the internal bond strength obtained was similar compared to the others resins previously synthesised and here presented. So, a small change in the synthesis parameters do not interfere on the final properties of particleboards.

CONCLUSIONS

The results obtained within the present research allowed: to understand the synthesis process for an UF resin and the manufacture of particleboards; the characterization methodology for resins and wood-based panels; and the identification of crucial factors for the synthesis. It also allowed to conclude that the % DMU is statistically significant for the internal bond. However, small changes in the synthesis parameters do not have a significant effect on the final properties of particleboards. These results were important for the company because it proves that small fluctuations, inherent to industrial synthetic process will not have a significant impact on the performance of the resin and consequently on particleboards properties. Furthermore, these results have also identified synthetic parameters that are worthy of fine tuning to yield better adhesive properties if required.

ACKNOWLEDGEMENT

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INSULATION BOARDS MADE OF ANNUAL AND PERENNIAL PLANTS BONDED WITH TANNINS AND OTHER ADHESIVES

Sebastian KRENN
Salzburg University of Applied Sciences, Forest Products Technology and Timber Construction Dept.
Markt 136A, 5431 Kuchl, Austria
Tel: 0043 5022 2100, Fax: 0043 5022 2099, E-mail: skrenn.htw-m2014@fh-salzburg.ac.at

Hermann HUBER
Salzburg University of Applied Sciences, Forest Products Technology and Timber Construction Dept.
Markt 136A, 5431 Kuchl, Austria
Tel: 0043 5022 2100, Fax: 0043 5022 2099, E-mail: hermann.huber@fh-salzburg.ac.at

Marius Cătălin BARBU
Transilvania University of Brașov, Faculty of Wood Engineering
Str. Universității nr. 1, 500068 Brașov, Romania
Salzburg University of Applied Sciences, Forest Products Technology and Timber Construction Dept.
Markt 136A, 5431 Kuchl, Austria
Tel: 0043 5022 2100, Fax: 0043 5022 2099, E-mail: cmbarbu@unitbv.ro

Alexander PETUTSCHNIGG
Salzburg University of Applied Sciences, Forest Products Technology and Timber Construction Dept.
Markt 136A, 5431 Kuchl, Austria
Tel: 0043 5022 2100, Fax: 0043 5022 2099, E-mail: alexander.petutschnigg@fh-salzburg.ac.at

Thomas SCHNABEL
Salzburg University of Applied Sciences, Forest Products Technology and Timber Construction Dept.
Markt 136A, 5431 Kuchl, Austria
Tel: 0043 5022 2100, Fax: 0043 5022 2099, E-mail: thomas.schnabel@fh-salzburg.ac.at

Abstract

The thermal insulation of buildings does not only saves energy costs, resources, but also helps to reduce CO_2 pollution. This study deals with the production of insulating boards based on biogenic raw materials and the investigation of their material properties. Maize stack and wheat straw as well as Miscanthus were used as the raw material for the boards. The binder used was a urea-formaldehyde resin, a sodium water glass and a tannin/hexamine system. The boards were produced by means of a hot pressing process under comparable conditions. In addition to the thermal conductivity of the boards, the particle distribution, the water uptake or release as well as the fire behavior of the different starting materials as well as of the resulting their panels were examined. The results show that the thermal conductivity of the panels from the raw materials used is in the range of natural insulation materials available on the market. The results from the water uptake test show low increase of the Miscanthus boards. The influence of the different adhesive systems plays only a subordinate role in the water uptake. However, differences in adhesives can be determined by the fire behavior test. Here, the tannin/hexamine binder shows advantages over the two other systems. Through these investigations, the potentials of plant based natural materials as an insulating material and as a binder were raised to provide a basis for further investigations.

Key words: *insulation board; maize stack; wheat straw; Miscanthus; fire behavior; thermal conductivity; water uptake.*

INTRODUCTION

Energy-efficient and easily recyclable buildings can be considered as a contribution to the reduction of greenhouse gas emissions. Nowadays petroleum-based plastic foams or mineral fibers are used for insulation purposes. However, a further increase in existing market shares and the extension of the use of renewable raw materials (e.g. annual or perennial plants) for insulations could have a positive effect on long-term CO_2 binding.

Building envelopes based on different straw products (e.g. straw bales) have a long tradition and are becoming increasingly popular (Ashoura et al. 2011), also because straw is a fast-growing

raw material, locally available and inexpensive. Beside these advantages, in the further processing of this construction material some specific properties should be considered (Nagl et al. 2015). Straw or straw bales have anisotropic material properties (Ashoura et al. 2011). This results in a great difference in the insulation performance depending on the mounting direction of the straw bale. Another consequence are the higher wall thicknesses, which should be considered already in the planning stage of the construction. These requirements can be taken into account for the new buildings, but in the case of refurbishment and renovation work on existing buildings due to increased wall thicknesses this can be a significant disadvantage compared to other construction or insulation materials. An alternative could be to produce straw-based insulating boards with standardized material properties and commercially available dimensions for the renovation area (Huber et al. 2015).

A problem that arose is given by the question related to the unfavorable bonding properties of wheat straw generated by its wax layer (Boquillon et al. 2004, Zhang et al. 2003). The works of Nagl (2014), Nagl et al. (2015), Krenn (2016), Krenn et al. (2017) serve as basis for this study.

OBJECTIVE

The aim of this research is to produce boards with densities lower than 300 kg/m³ from different mechanically shredded materials from agricultural annual and perennial plants with adhesives based on renewable materials, e.g. tannin (Pichelin et al. 2006, König 2006, König and Roffael 2002). Further, for different types of boards, beside the thermal conductivity important parameters were recorded e.g. water absorption and the fire behavior, then assessed or compared.

MATERIAL, METHOD, EQUIPMENT

For the tests were used stems of corn (*Zea mays* L.), elephant grass (*Miscanthus spec.*) and wheat (*Triticum* L.). The materials were harvested and air-dried using commercially available agricultural machines. The corn stalk parts were not extra processed. The Miscanthus was further reduced to particles with a garden shredder. The wheat straw was mulched with agricultural equipment. This resulted in different particle sizes for the three raw materials (Fig. 1).

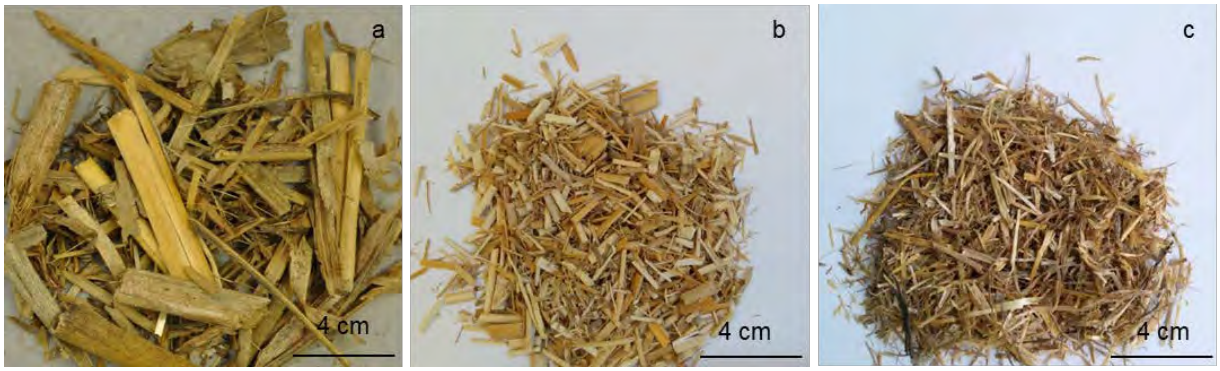

Fig. 1.
Raw material from annual and perennial plants a) maize stalk b) miscanthus and c) wheat stalk

For the bonding of straw particles, was used a tannin adhesive (mimosa type). Stirring of the tannin binder was made with 50 parts by weight of powder and water using a laboratory mixer. In the next step, the solution was adjusted to pH 8.5 with a 10% sodium hydroxide solution (using a pH meter). Shortly before gluing, 3% hexamine was added to the solution as a hardener.

In order to compare the results, 10F102 urea-formaldehyde resin (UF) from Methadynea Group with a solids content of 66% and an ammonium sulfate solution as hardener were used, and sodium bicarbonate from the Merk Group with a solid content of 50% was used as the third glue.

The particle size distribution of the raw material was carried out by means of a vibrating or vibrating-screening method according to EN 15149-1 (2011) and -2 (2011). The particles were graded in decreasing size classes with the sieving machine AS 200 (Retsch) with a shaking time of 15 minutes and an amplitude of 50. The particle size ≥ 2 mm was used for the further manufacturing of the material to the insulation board.

The insulation boards were produced with a dimension of 450 mm × 450 mm × 25 mm and three different adhesive types were used for the tests. For all laboratory boards was used 15% adhesive, based on the absolutely dry mass of the particles. For the tannin and urea formaldehyde (UF) glues, 3% hardener were added.

The simultaneous mixing and gluing was carried out in a ploughshare mixer. Consequently, the glued stalks, straw sticks or fibers were manually scattered in a wooden forming box. The pressing parameters - temperature of 100°C, cycle and the time - were kept constant for all production tests (Tab. 1). The target thickness for the insulation panels was 25 mm.

Table 1

Press program of the manufactured insulation panels

Process step	Board thickness (mm)	Press time (s)
1	23	300
2	28	30
3	25	300
4	26	60
5	28	60
6	250	10

The thermal conductivity was measured according to EN 12667 (2001) with the λ-Meter EP500e from Lambda-Messtechnik. The λ-value was determined at 10°C, 25°C and 40°C.

For the water uptake the samples were cut in the format 80 mm x 80 mm x 25 mm. This test was carried out according to EN 1609 (2013). The first measurement was performed after 12 hours. The samples were removed from the vessel, drained/wiped and weighed. The second measurement took place after 24 hours, followed by the drying of the samples from various types of straw (maize stalks, wheat stalks and Miscanthus) at 50°C. During drying, the insulation panels were weighed at regular intervals. Drying was continued until the samples regained the initial weight.

The loss of mass as an effect of fire with a Bunsen burner was carried out with a self-developed experimental construction (Fig. 2). In this case, the variable loads of the equipment (e.g. Bunsen burner), which influence the weight measurement, were not taken up with the balance, but were measured with an external load, so only the weight of the sample (40 mm × 40 mm) and its holder on the balance were measured.

Fig. 2.
Own experiment setup and detailed view for determining the mass loss during exposure to fire.

RESULTS AND DISCUSSION

The raw materials from plants used for this study differ in the composition of the particle distribution (Fig. 3). Especially the maize stalks have less fine particles. The difference to the other types of straw can be explained by the mechanical processing during harvesting. The wheat straw was pressed with an intent shredder to form large square bales and mulched for the experimental boards. By these processing, a higher content of fine particles compared to maize cannot be avoided. The Miscanthus stalks were crushed with a shredder. The particle distributions of the wheat straw and Miscanthus stalks are comparable. The largest proportion in the fraction of > 4 mm is the maize stalk, since it has been processed less intensively. As a source material for the boards manufacture and further tests were used only the particles > 2 mm (Krenn et al. 2017).

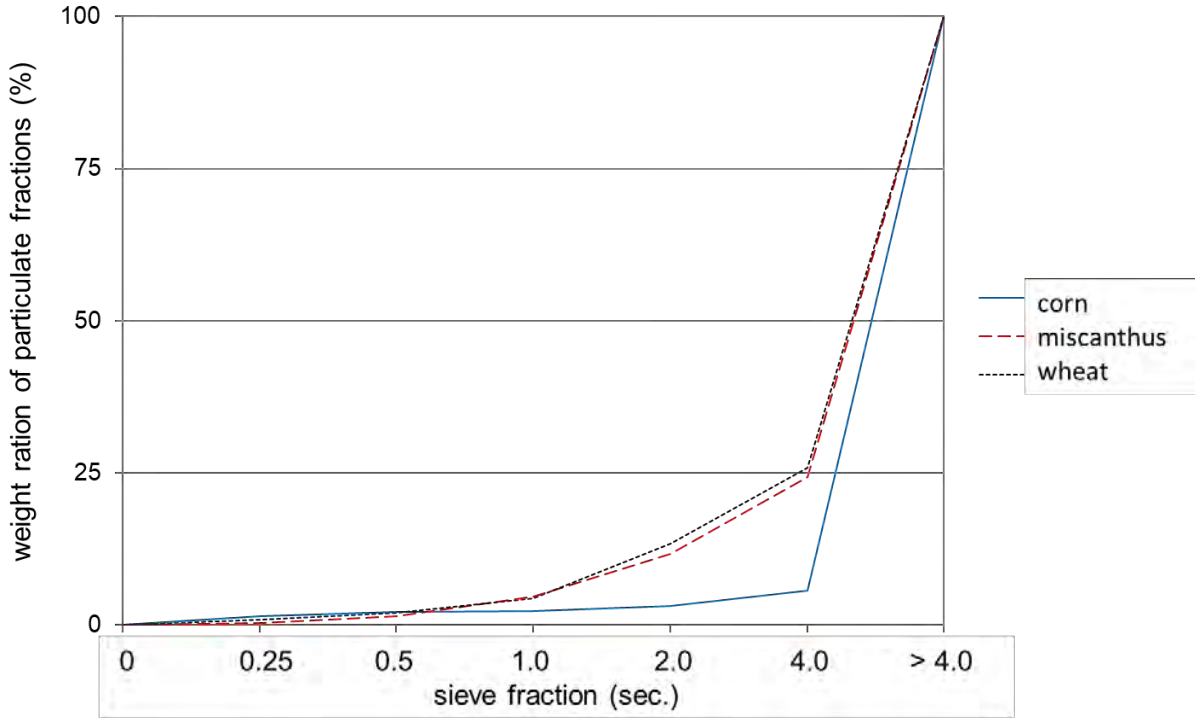

Fig. 3.
Weight parts of the residues from different plants per particle fraction.

The manufacture of the insulation boards from various vegetable raw materials and adhesives took place without difficulties. In addition to the UF adhesive used by Nagel et al. 2015 for their experiments, a tannin mixture and water glass were used as glues. Before testing the thermal conductivity, water absorption and fire behavior, the insulation boards were stored in a normal climate at a temperature of 20°C and a relative air humidity of 65% until the constant weight was achieved.

Figure 4 shows the thermal conductivity of the analyzed boards as a function of the bulk density. The spans of the raw density from maize stalks (80 – 300 kg/m³) or wheat straw (50 – 300 kg/m³) have higher values than the boards from Miscanthus (160 – 300 kg/m³). This can be explained by the low bulk densities of the two materials (Nagl et al. 2015, Krenn et al. 2017). In addition, it can be observed a linear dependence of the thermal conductivity on the density of the different boards in the tested area. Dimensionally stable insulation boards could be produced with raw densities around 80 kg/m³ from maize stalks. For the boards based on other raw materials, this low bulk density could not be achieved. In addition to the proper low thermal conductivity in a possible application as an insulation material, further material properties such as water absorption and fire behavior were investigated. For these tests, were produced boards with a bulk density of 250 kg/m³, in order to ensure a sufficient comparability of the different raw materials.

Figure 5 shows the water uptake and water release of the wheat straw board (250 kg/m³) manufactured with different adhesives. After 24 hours, the water absorption is 270-280% when using UF and water glass (WG) as binder. The value of the tannin-bonded boards (Ta) is 250%. The highest percentage of water absorption was recorded for the wheat straw boards bonded with UF. When looking at the actual water absorption as a function of the sample surface, the findings are relativized. The measured values of the boards bonded with water glass are with 38,63 kg/m² in front of the samples produced with UF (39,53 kg/m²) and tannin (44,52 kg/m²). Similar values were confirmed by the results of Nagl (2014). After 24-hour water immersion the samples were re-dried in oven at 50° C. After a drying time of 69 hours, all insulation boards made of straw had reached their starting weight again. No significant differences in the drying times of the boards bonded with different glues were found. The graphs of water absorption and water release of the various boards are comparable. The adhesive used here shows only a small influence on these properties.

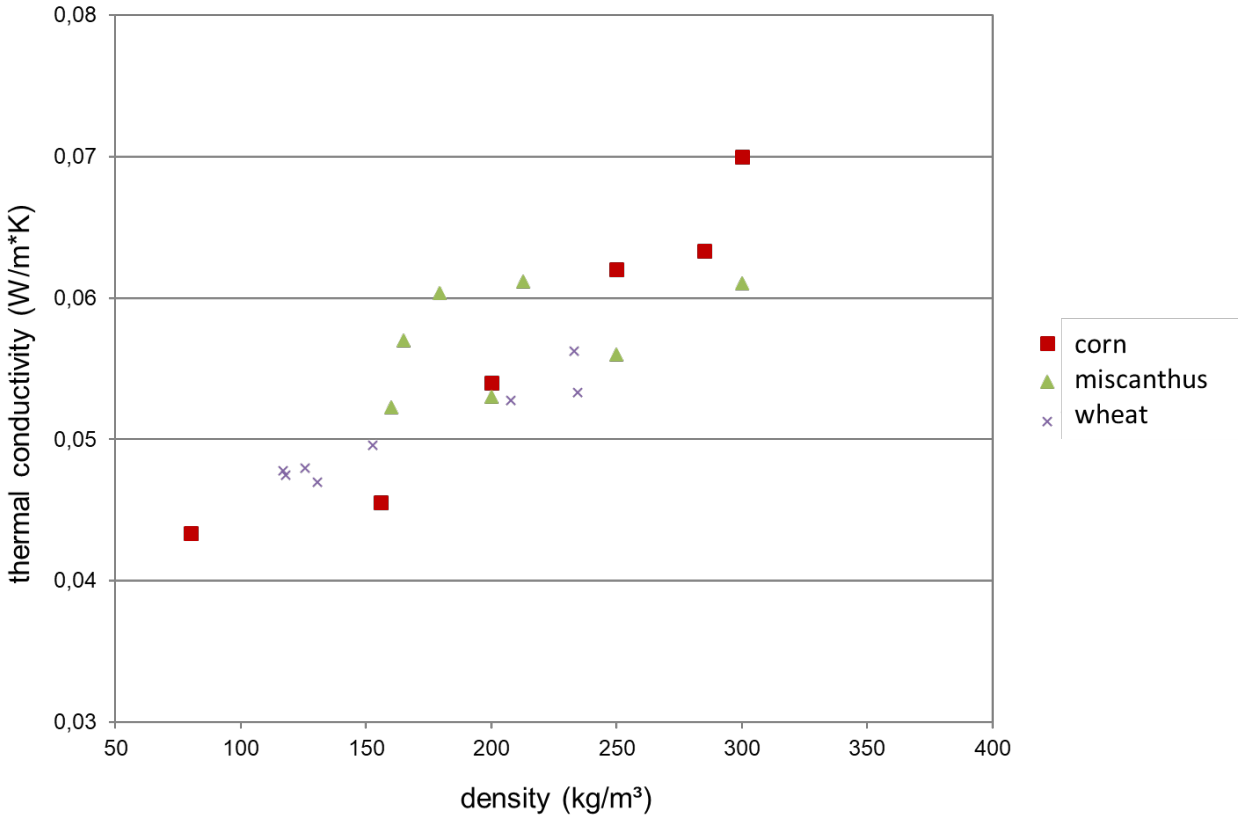

Fig. 4.
Thermal conductivity of the tested boards with tannin adhesive (Ta) as a function of the raw density at a temperature of 10° C and a temperature difference of the measuring plates of 15 K.

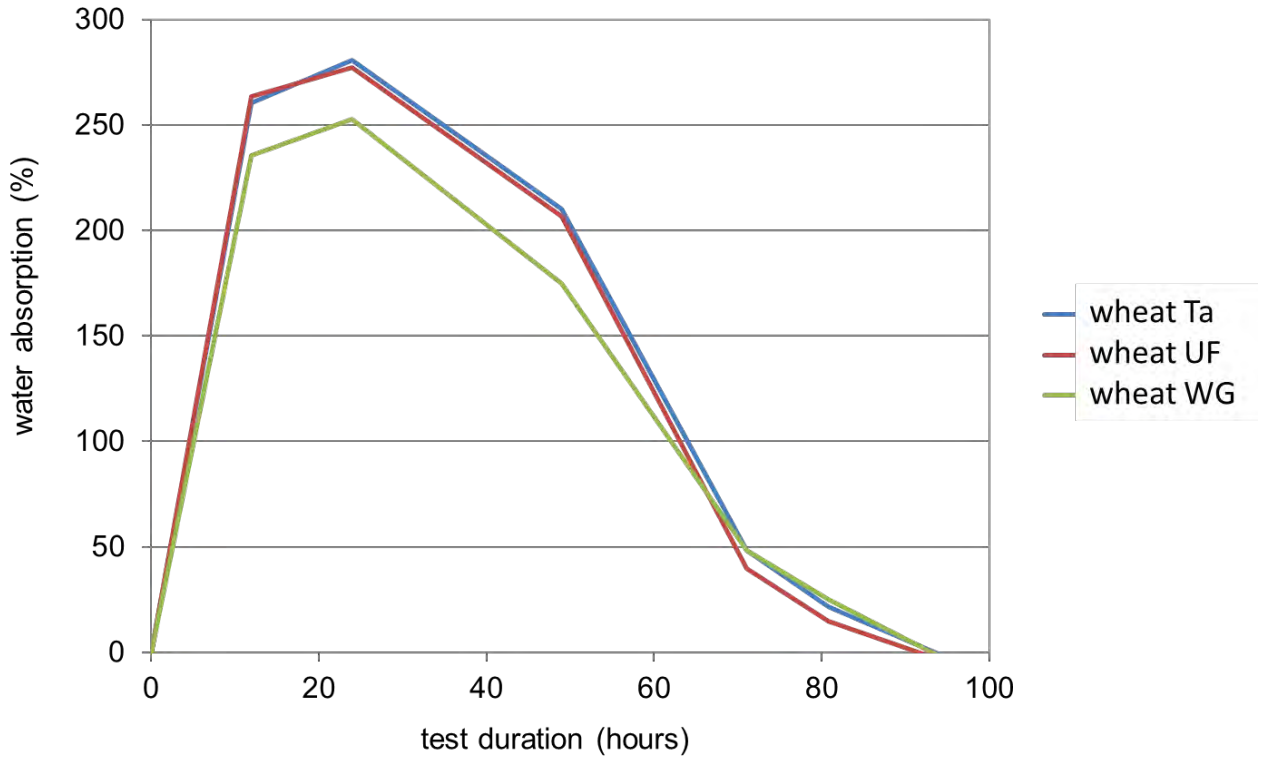

Fig. 5.
Influence of different adhesives on wheat straw boards (250 kg/m³) on the water absorption and water release.

Figure 6 shows the water absorption and water release of different boards made of maize stalks, Miscanthus and wheat straw. The values of the total water absorption of the laboratory boards after 24h water immersion were for wheat 44.52 kg/m², corn 41.88 kg/m² and Miscanthus 25.76 kg/m².

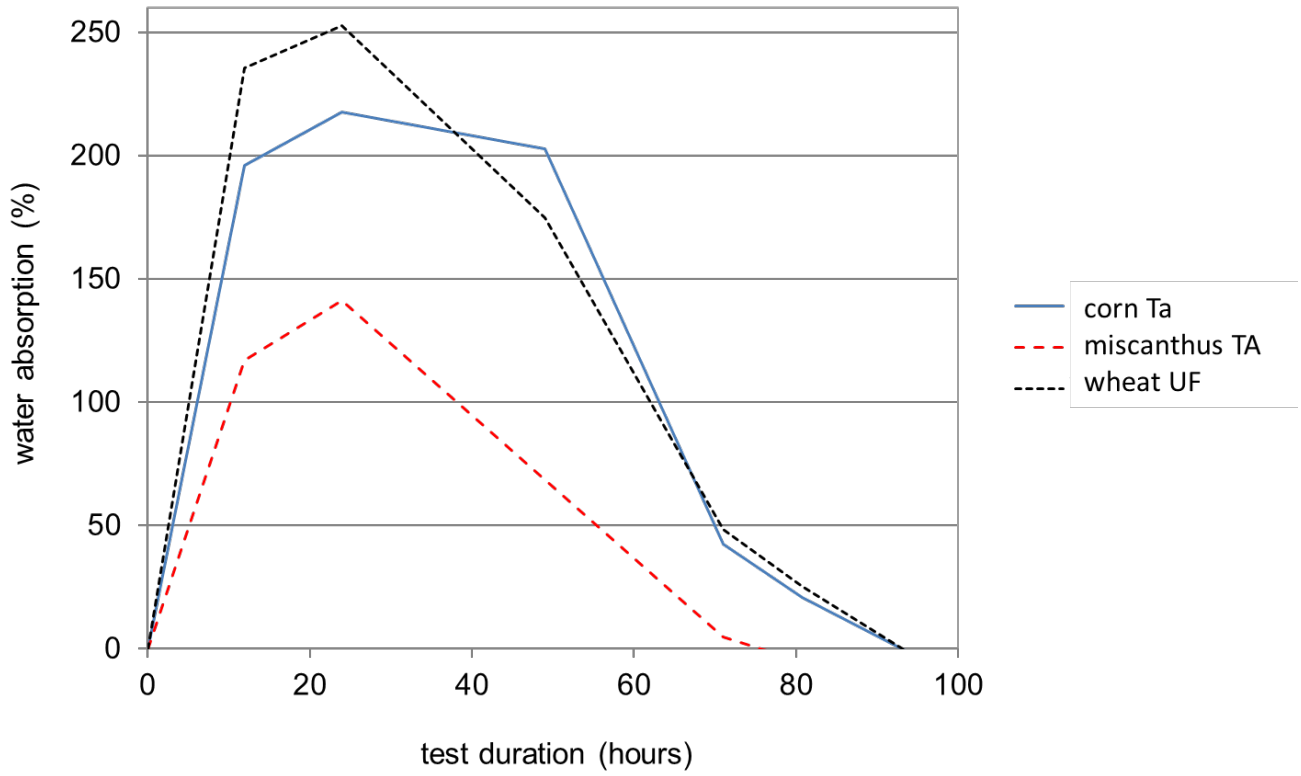

Fig. 6.
Influence of the different insulation boards (250 kg/m³) from maize stalk, miscanthus and wheat straw bonded with tannin-hexamine (Ta) on the water absorption and water release.

The maize stalk and wheat straw dried very uninform. Miscanthus showed the lower percentage for water absorption. This behavior has also been described by Nagl et al. (2015) in the water uptake of loose miscanthus stalk components. The highest value was about 140% (or 25.76 kg/m²). Miscanthus also dried most uniform. The boards of Miscanthus and maize stalks were dimensional stable after water absorption and drying. After the water uptake, the boards of wheat straw fell apart into larger parts.

In addition to water absorption, the inadequate fire behavior of insulating panels made from natural raw materials is often mentioned as a possible restriction to the application. In the present study the fire behavior of tannin-based insulation boards was investigated and compared with boards bonded with a standard adhesive for the wood industry.

Figure 7 shows the fire behavior of wheat straw boards (250 kg/m³) as mass changes of the materials bonded with the as tannin glue (Ta), urea formaldehyde resin (UF) and water glass (WG). After small mass losses and stable values, the boards were continuously reduced in weight by the established end of test, after 5 minutes. The small decrease after 30 seconds can be attributed to the warming of the sample (Kollmann 1960). The stagnating value up to the test time of 60 seconds is caused by the evaporation of the free and bounded water because, during this process, the temperature in the material is not increased and remains constant within a range of 102 ± 2°C (Lingens 2003). At this temperature, there is no material degradation, but only a loss due to the dehydration of the material. In the further test steps, a steady decrease of the mass by thermal degradation was recorded. After 5 minutes of fire load, the insulation board with wheat straw bonded with tannin has a higher mass than the similar boards produced with the other two types of adhesive (UF and WG). The mean value at the end of the test is still 38.3 % of the starting mass for the wheat straw-based insulation boards bonded with tannin glue. For the boards glued with UF, 24.8% of the initial mass is still 23.1% of the initial weight for the insulation panels with water glass.

A similar tendency of the mass reduction could be found in the comparison between the boards with the following raw materials: maize stalks, wheat straw and Miscanthus with tannin (Ta) as a binder (Fig. 8). At the end of the test period of 5 minutes, the mean value of the maize stalk boards

is still 44.4% of the initial mass. In the case of the wheat straw insulation boards, the remaining end weight is still 38.3 % of the starting weight and the Miscanthus insulation boards are 38.5 % of the initial mass.

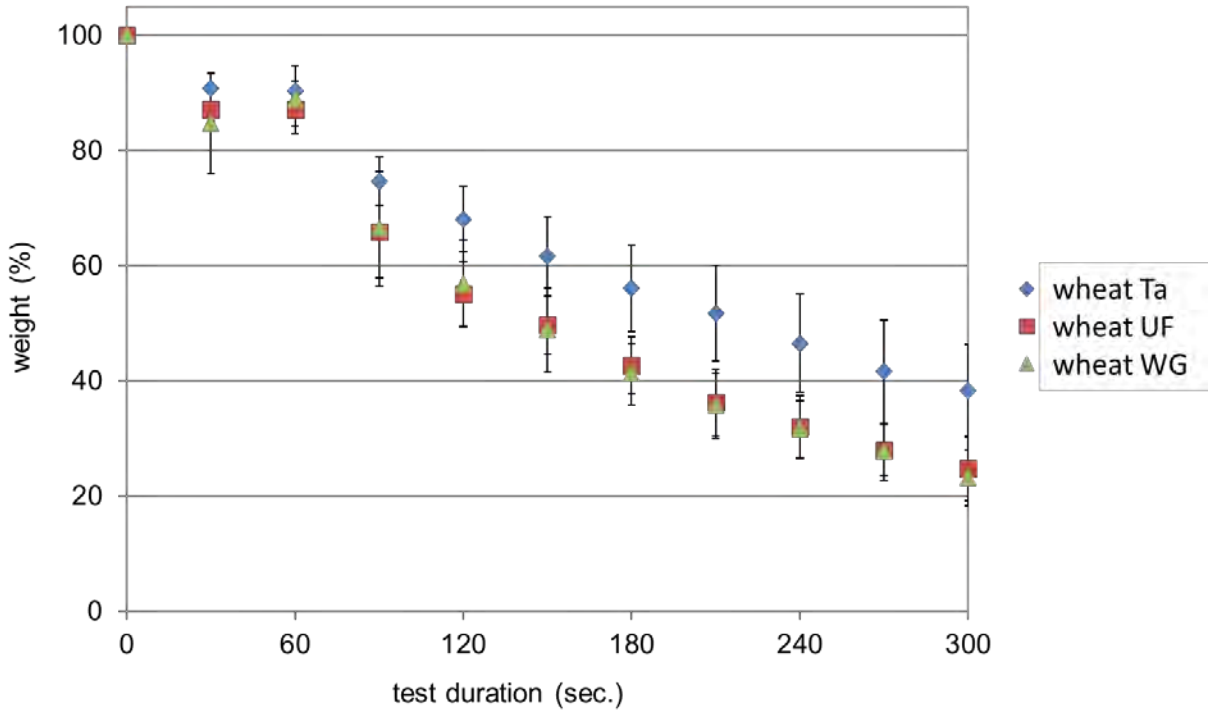

Fig. 7.
Mass change after fire test of wheat straw boards (250 kg/m³) with 3 different types of adhesive.

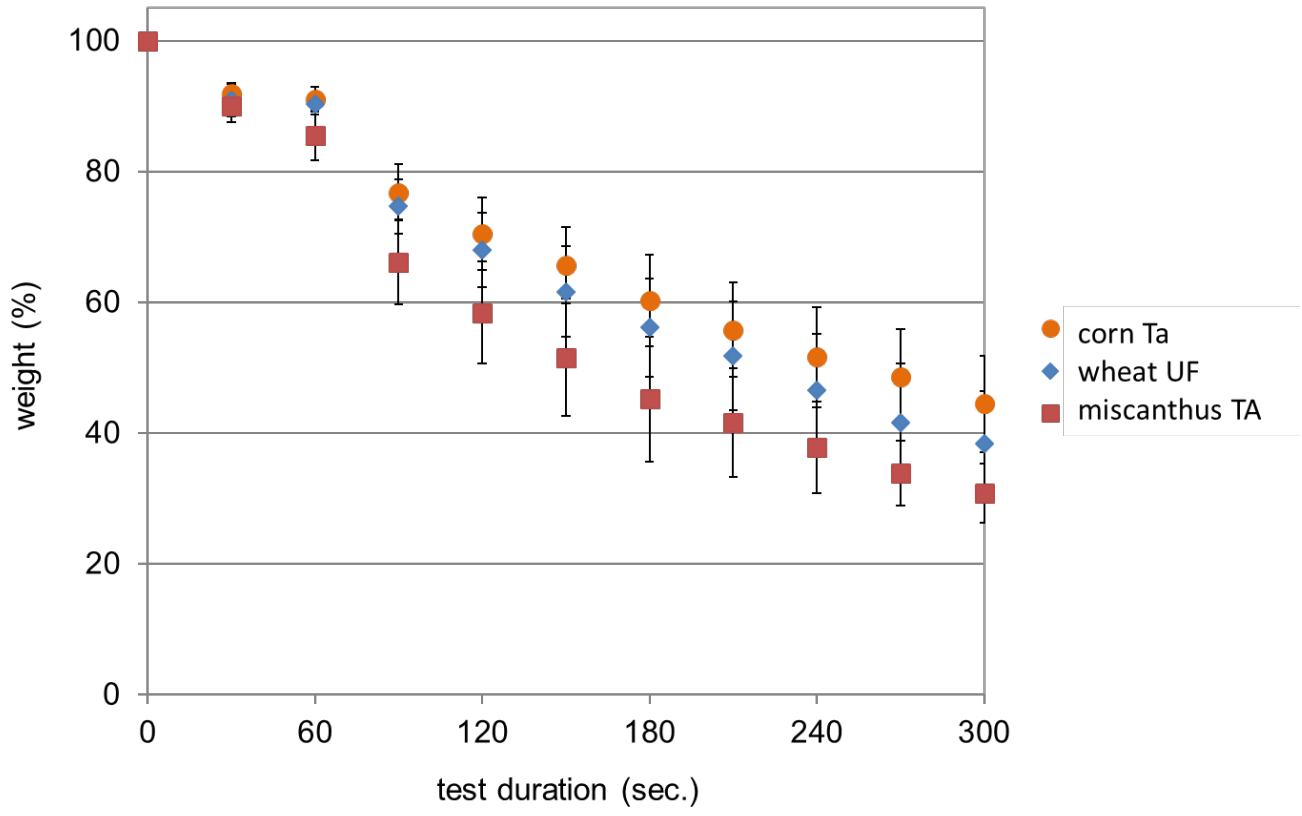

Fig. 8.
Mass change of the various insulation boards (250 kg/m³) during fire test.

Figure 9 shows the visual changes of the different insulation boards at the end of fire exposure. The maize stalk board is burned to two-thirds after completion of the test. After 300 seconds, the entire underside glowed and was completely burned. Only the leaves and stems of the corn plant were still present to some degree. Regarding the boards made of Miscanthus, after completion of the tests were recognizable only ash and some intact stems. When the specimen was removed from the test site, only individual fragments could be grabbed by hand. This rapid combustion is due to the difficult adhesion of Miscanthus. Due to the poor uptake or wetting of adhesives, the adhesion in the board is unstable under the influence of fire, so individual parts can fall apart. The wheat straw insulation board was completely carbonized after completion of the test. Furthermore, some stems also unfasted during the test. After completion of the tests, the test specimens were no longer adequately glued and fell apart when the test device was removed.

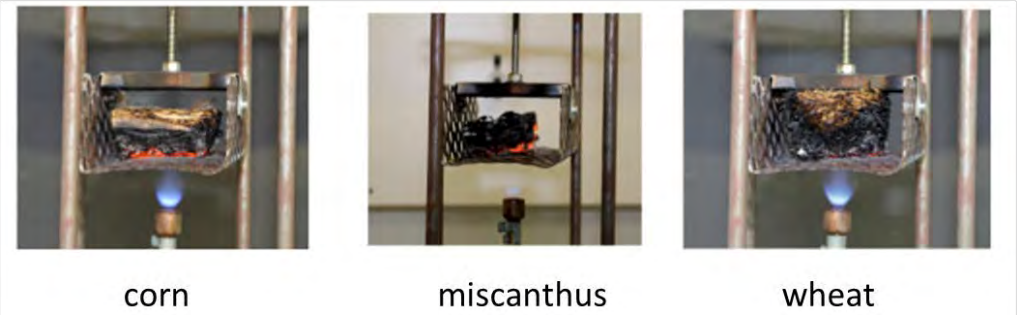

Fig. 9.
Visual changes of the insulation boards after the 300 seconds of the fire test

CONCLUSIONS

The main focus of these investigations and the results presented here were based on the research question about the feasibility of insulating boards made of plants by means of tannin-hexamine bonding. By using different adhesive systems, the comparability of natural adhesive systems with a standard glue could be demonstrated in the results of the water absorption or water release. This depends less on the different adhesive system, but on the raw material used. The results show the lowest water absorption for Miscanthus. However, the significant good results could not be attributed to the fire behavior. Here, the maize stalk-based boards showed the least mass loss. However, it is notable that the tannin adhesive protects the insulating boards against the effects of fire at laboratory scale. Further studies are still necessary to implement the positive research results so far for applications in the construction sector.

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INDUSTRIAL RESEARCH CONCERNING THE SIMULTANEOUS REDUCTION OF DENSITY AND FORMALDEHYDE EMISSION OF PARTICLEBOARDS

Karoly LENGYEL
KASTAMONU Romania
Str. Ierbuş nr. 37, 545300 Reghin, Romania
E-mail: Karoly.Lengyel@kastamonu.ro

Mihaela CÂMPEAN
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: campean@unitbv.ro

Abstract

The outcome of the present research was to find a cost-effective solution for reducing the density and formaldehyde emission of particleboards without significantly affecting the other important properties of the boards. Twelve different recipes, obtained by varying the density, the percentual participation of softwood / hardwood chips and the percentual participation of an isocyanate-based additive, were used to manufacture 200 pieces of particleboards of each recipe at industrial scale. The density, formaldehyde emission, swelling in thickness, surface absorption, bending strength and modulus of elasticity in bending, internal bond and surface soundness were determined for each recipe and then compared in order to establish the optimum variant, namely the one which allows reducing simultaneously the density and the formaldehyde emission without lowering the other physical and mechanical performances of these boards.

Key words: *particleboards; density; formaldehyde emission; bending strength; modulus of elasticity; internal bond.*

INTRODUCTION

The present trend in particleboard industry is to produce boards with as low as possible amount of wooden raw material, with low density and very low formaldehyde emission, which should also meet all existing quality standards and which should not increase the production costs, in order to maintain the price-advantage of particleboards in the competition with other wood-based panels.

Different weight-saving techniques were explored worldwide. Using light available wood species like bamboo or poplar (*e.g.* Akrami *et al.* 2014; Malanit *et al.* 2010), combinations between wood chips and agricultural fibres (*e.g* rice straw - Akyldiz *et al.* 2015; cotton – Guler *et al.* 2001; kenaf - Kalaycıoğlu and Nemli 2006; sunflower stalks – Bektas *et al.* 2005 etc.), increased resination (*e.g.* Monteiro *et al.* 2016) and optimized board density profiles (*e.g.* Lüdtke *et al.* 2007, Michanickl 2006, Poppensieker and Thömen 2005) are only four of the directions followed in international research. Each technique also has its downsides: either the production of lightweight panels is too laborious (involving high costs), or the mechanical and physical performances of the panels are significantly decreased (Barbu 2015).

Thus, a cost-effective solution for producing particleboards with reduced density, reduced formaldehyde emission and at least standard-according physical and mechanical properties, still remains a challenge of the particleboard industry today. The present research is an attempt in this direction.

OBJECTIVE

The main objective of the present research was to test different composition recipies of particleboards by varying the density of the boards, the percentage of hardwood / softwood chips and also by varying the additive amount. The final outcome was to find optimum composition recipies which allow simultaneous reduction of the density and of the formaldehyde emission of the boards, without affecting other important physical and mechanical properties, such as the swelling in thickness, the surface absorption, the bending strength and the modulus of elasticity in bending, the internal bond, the surface soundness etc.

MATERIAL, METHOD, EQUIPMENT

The material used within the present research consisted in low-emission P2 raw particleboards with thickness of 28mm, produced by KASTAMONU Romania.

First, particleboards with normal density (ρ=625 kg/m^3) were tested, then particleboards with the density reduced by 7%. These two tests were called „base tests" because the boards contained no additive, thus serving as reference. The third set of tests was meant to improve the properties of the boards with reduced density by using an isocyanate additive into the adhesive's recipe. Two percentages of additive were tested: 0.25% and 0.4%.

By varying also the hardwood/softwood percentage of chips within the raw material recipe, twelve combinations resulted (Table 1). A number of 200 particleboards from each recipe were produced.

Table 1

Composition recipe of the 28mm thick particleboards

Ref. Nr.	Percentual participation of hardwood / softwood chips	Percentual participation of the isocyanate additive, %	CL adhesive, %	SL adhesive, %	CL urea, %	SL urea, %	CL hardener, %	SL hardener, %
Particleboards with normal density (ρ=625 kg/m^3)								
1	25/75	No additive	8.0	11.0	0.00	0.17	0.22	0.18
2	30/70	No additive	8.0	11.0	0.00	0.17	0.22	0.15
3	35/65	No additive	8.0	11.0	0.00	0.17	0.22	0.20
Particleboards with 7% reduced density (ρ=580 kg/m^3)								
4	25/75	No additive	8.0	11.0	0.00	0.17	0.22	0.18
5	30/70	No additive	8.0	11.0	0.00	0.17	0.22	0.18
6	35/65	No additive	8.0	11.0	0.00	0.17	0.22	0.18
7	25/75	0.25	8.0	11.0	0.00	0.17	0.18	0.22
8	30/70	0.25	8.0	11.0	0.00	0.17	0.18	0.22
9	35/65	0.25	8.0	11.0	0.00	0.17	0.18	0.22
10	25/75	0.4	8.0	11.0	0.00	0.17	0.22	0.18
11	30/70	0.4	8.0	11.0	0.00	0.17	0.22	0.18
12	35/65	0.4	8.0	11.0	0.00	0.17	0.18	0.22

CL = core layer

SL = surface layer

The applied technological pressing parameters were the following:

- feed speed of the press: 230mm/s;
- press factor: 5.95s/mm
- maximum temperature: 245°C;
- maximum pressure: 3.12N/mm^2.

After pressing, the boards were conditioned for 30 minutes at ambiental temperature and then, specific test pieces were cut out of a randomly selected board, according to EN 326-1:1994 (confirmed 2014), in order to determine the most relevant physical and mechanical properties for particleboards.

Density

The particleboard density was determined according to EN 323:1993. Twenty samples from each recipe, sized at 50mm x 50mm x 28mm, were first weighed at an accuracy of 0.01g and then measured at an accuracy of 0.1mm. The thickness (*t*) was measured in the central point by means of a micrometer and the dimensions b_1 and b_2 were measured at mid width and length, according to Fig. 1, by means of a sliding gauge.

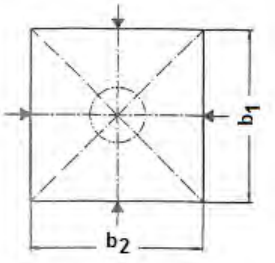

Fig.1.

Measuring the dimensions of the particleboard samples for density determination.

The density of each samples was then calculated according to the relation:

$$\rho = \frac{m}{b_1 \cdot b_2 \cdot t} \cdot 10^6 [kg / m^3] \quad (1)$$

where: m is the sample mass, in g;
b_1, b_2 – sample dimensions, in mm;
t – sample thickness, in mm.

Formaldehyde emission

The flask method (EN 717-3:1996) was used for the determination of the formaldehyde emission. Twelve test pieces from each recipe were used to determine the moisture content, according to EN 322:1993. Other twelve test pieces from each recipe, sized at 25mm x 25mm x 28mm, were first weighed, then attached to the inner side of the lid of a glass container by means of a hook as shown in Fig. 2, above a bath of 50ml distilled water. Two glass containers were used, for six replicates each.

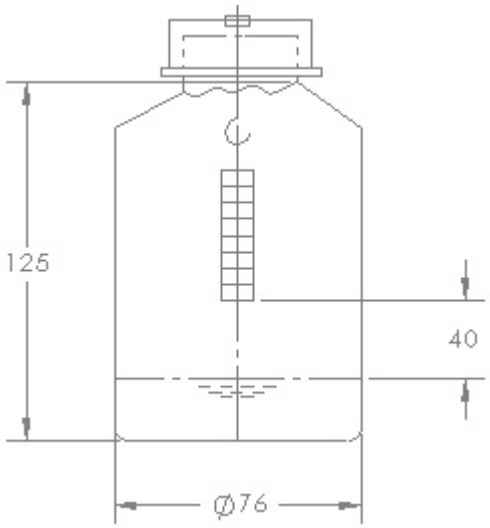

Fig. 2.
Principle of determining the formaldehyde emission from particleboards by means of the flask method.

The bottles were closed completely airtight and maintained at constant temperature (40±1)ºC for (180±1) minutes in a Lange-LT200 thermoreactor (Fig. 3). During this time, the formaldehyde released by the particleboard samples was absorbed into the water. Then the formaldehyde content of the water was determined photometrically by the acetylacetone method. To this purpose, the 50 ml of solution from each glass container were transferred immediatly after the (180±1) minutes into two 50ml flasks and let to cool and ambient temperature to 20°C. 10ml from this aqueous solution were taken by means of a pipette and added to 10ml acetylacetone solution and 10ml ammonium acetate solution in a 50ml flask. The flask was stoppered, shaken and warmed for 15 minutes in a water bath at (40±1)ºC. Then the formaldehyde absorbed in these 10ml of solution from the containers was determined by means of a Lasa 30 spectrophotometer (Fig, 3).

The amount of formaldehyde emission according to the flask method was calculated according to the formula:

$$F_v = \frac{(A_s - A_B) \cdot f \cdot 50 \cdot 10 \cdot (100 + H)}{m} \left[\frac{mg}{kg}\right] \quad (2)$$

where:
A_s is the absorbance of the analysed solution from the glass bottle;
A_B - absorbance of an analysis with distilled water;
f - slope of the calibration curve, in mg/ml;
H - moisture content of the test piece, in %;
m - mass of the test piece mass, in g.

Fig. 3.
Equipment for the determination of the formaldehyde emission by the flask method.

Swelling in thickness after water immersion

The swelling in thickness after water immersion was determined according to EN 317:1993. Eight samples from each recipe, sized at 50mm x 50mm x 28mm, were first measured in thickness (at the intesection of diagonals) by means of a micrometer at an accuracy of 0.01mm. Then they were vertically immersed for 2 hours in a clean, still, thermostatically controlled water bath having a pH of 7±1 and a temperature of (20±1)°C (Fig. 4). During the test, the test pieces were separated from each other and from the bottom and the sides of the water bath. The test pieces were immersed so that their upper edges were covered by (25 ± 5) mm of water throughout the test. The water was changed after each test.

After 2 hours of immersion, the test pieces were taken out of the water and, after removing the water in excess, the thickness of each test piece was measured again.

The swelling in thickness (G_t) was expressed according to the following formula:

$$G_t = \frac{t_2 - t_1}{t_1} \cdot 100[\%] \qquad (3)$$

where: t_2 is the test piece thickness after immersion, in mm;

t_1 – test piece thickness before immersion, in mm.

Fig. 4.
NUVE-BS402 equipment used for the determination of the swelling in thickness after water immersion of the tested particleboards.

Surface absorption

The surface absorption was determined according to EN 382-1:1993. Eight samples from each recipe, sized at (300±2)mm x (300±2)mm x 28mm, were placed on a (60±5)° inclined support (Fig. 5). The pipette for dropping the toluene sollution was placed at a distance of 1±0.1mm, perpendicular to the panel surface. 1 ml of toluene was dropped at regular time intervals of 4s and left to flow on the panel surface. The maximum length of the trace was measured along a line parallel to the test piece margins, at an accuracy of ±1mm. The operation was repeated on the other panel face as well. The surcae absorption (A_s) was then calculated as arithmetic mean of the two measured values, in mm.

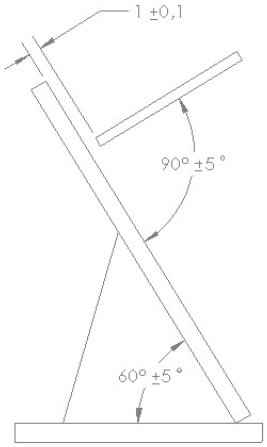

Fig. 5.
Experimental set-up for the determination of the surface absorption: 1-pipette; 2-test piece; 3-inclined support.

Bending strength and modulus of elasticity in bending

The bending strength and modulus of elasticity in bending were determined according to EN 310:1993. Six samples from each recipe, sized at (20*t*+50)mm x 50mm x panel thickness (*t*), were tested by means of the IMAL IB600 equipment (Fig. 6).

The distance between the centers of the supports was adjusted at 20 times the nominal thickness of the panel. The test piece was placed flat on the supports, with its longitudinal axis at right angles to those of the supports with the center point under the load. The load was applied at a constant rate of the cross-head movement throughout the test. The rate of loading was adjusted so that the maximum load was reached within (60±30)s. The deflection in the middle of the test piece (below the loading head) was measured to an accuracy of 0.1mm. These values were plotted against the corresponding loads measured to an accuracy of 1% of the measured value. The maximum load was recorded to an accuracy of 1% of the measured value.

Fig. 6.
IMAL IB600 equipment used for the determination of the bending strength and modulus elasticity in bending of the tested particleboards.

The bending strength of each test piece (f_m) was calculated according to the formula:

$$f_m = \frac{3 \cdot F_{\max} \cdot l_1}{2 \cdot b \cdot t^2} [N/mm^2] \quad (4)$$

where: F_{max} is the maximum load, in N;
l_1 – distance between the centers of the supports, in mm;
b – test piece width, in mm;
t – test piece thickness, in mm.

The modulus of elasticity in bending (E_m) was calculated according to the formula:

$$E_m = \frac{l_1^2 \cdot (F_2 - F_1)}{4 \cdot b \cdot t^2 \cdot (a_2 - a_1)} [N/mm^2] \quad (5)$$

where: ($F_2 - F_1$) is the increment of load on the straight line portion of the load-deflection curve;
($a_2 - a_1$) – deflection increment at the mid length of the test piece corresponding to ($F_2 - F_1$).

Tensile strength perpendicular to the plane of the board (internal bond)

The tensile strength perpendicular to the plane of the board, also called internal bond, was determined according to EN 319:1993. Eight samples from each recipe, sized at 50mm x 50mm x 28mm, were tested by means of the IMAL IB600 equipment (Fig. 6). Each test piece was loaded with a tension force uniformly distributed perpendicular to its surface until rupture occurred.

The tensile strength (f_{t1}) was calculated according to the formula:

$$f_{t1} = \frac{F_{\max}}{a \cdot b} [N/mm^2] \quad (6)$$

where : F_{max} is the maximum force, in N;
a, b – test piece length andd width, in mm.

Surface soundness

The surface soundness was determined according to EN 311:2002. Eight samples from each recipe, sized at 50mm x 50mm x panel thickness, were tested by means of the IMAL IB600 equipment (Fig. 6).

A steel mushroom-shaped pad with a diameter of 35.6±0.1 mm is glued by means of a hot-melt adhesive on the sample surface. A circular groove is cut through the coating material so that it just breaks through into the underlying board.

The surface soundness (SS) is calculated from the tensile load required to pull off a defined surface area:

$$SS = \frac{F}{A} [N/mm^2] \quad (7)$$

where: F is the maximum force recorded by the equipment, in N;
A – surface area delimited by the groove (1000mm^2).

RESULTS AND DISCUSSION

The results concerning the influence of the percentual participation of hardwood / softwood chips in the composition recipe upon the properties of particleboards with normal and reduced density, respectively, are presented in Tables 2 and 3.

Table 2

Influence of the percentual participation of hardwood / softwood chips in the composition recipe upon the properties of normal density particleboards

Raw material recipe (hardwood/softwood)	ρ, kg/m^3	F_v, mg/kg	G_t, %	A_s, mm	f_m, N/mm^2	E_m, N/mm^2	f_{t1}, N/mm^2	SS, N/mm^2
25/75	618.1	729.7	7.1	34.6	11.1	2434	0.35	1.09
30/70	625.5	723.3	9.1	37.7	10.6	2209	0.36	1.05
35/65	629.1	703.7	10.2	40.9	11.2	2437	0.37	0.98

Reducing the percentual participation of softwood chips in favour of the hardwood chips in the composition recipe of particleboards with normal density lead to the:

- increase by 17.8% of the density;
- decrease by 3.5% of the formaldehyde emission;
- increase by 43.6% of the swelling in thickness;
- increase by 18.2% of the surface absorption;
- insignificant modification of bending strength, modulus of elasticity and tensile strength;
- increase by 3.9% of the surface soundness.

By analysing the property modifications brought by the raw material participation in the particleboard recipe, the best variant was considered the one with lowest percentage of hardwood chips (25/75), which is also the most cost-efficient one.

Table 3

Influence of the percentual participation of hardwood / softwood chips in the composition recipe upon the properties of particleboards with reduced density

Raw material recipe (hardwood/softwood)	ρ, kg/m^3	F_v, mg/kg	G_t, %	A_s, mm	f_m, N/mm^2	E_m, N/mm^2	f_{t1}, N/mm^2	SS, N/mm^2
25/75	589.8	1061.6	10.8	46.7	8.8	2252	0.23	1.00
30/70	589.8	959.8	14.3	75.0	9.1	2156	0.25	0.95
35/65	590.9	901.6	12.2	71.3	9.6	2215	0.24	0.99

Reducing the density of the particleboards by 7% had negative influence upon all properties of the particoleboards:

- the formaldehyde emission increased;
- the swelling in thickness and surface absorption increased majorly;
- bending strength, modulus of elasticity and tensile strength decreased;
- the surface soundness decreased.

Consequently, this is not a viable alternative and in order to improve the properties at the reduced density particleboards, the use of an additive in the composition recipe is absolutely necessary. Two percenatges of the P-MDI (isocyanate) additive were tested, namely 0.25% (Table 4) and 0.4% (Table 5).

Table 4

Influence of the introduction of a P-MDI additive (0.25%) in the composition recipe of particleboards with reduced density upon their properties

Raw material recipe (hardwood/softwood)	ρ, kg/m^3	F_v, mg/kg	G_t, %	A_s, mm	f_m, N/mm^2	E_m, N/mm^2	f_{t1}, N/mm^2	SS, N/mm^2
25/75	593.2	569.3	8.4	40.3	12.3	2186	0.26	1.19
30/70	597.0	495.0	7.3	35.0	11.6	2101	0.28	1.17
35/65	590.2	540.3	13.2	49.6	12.2	2396	0.29	1.06

By adding 0.25% P-MDI isocyanate additive into the composition recipe of particleboards with reduced density, the following property modifications were achieved compared to the particleboards with reduced density without additive (the raw material recipe 25/75 was considered as reference):

- the formaldehyde emission decreased by 46.37%;
- the swelling in thickness decreased by 22.22%;
- the surface absorption decreased by 13.70%;
- the bending strength increased by 39.77%;

- the modulus in elasticity decreased by 2.93%;
- the tensile strength increased by 13.04%;
- the surface soundness increased by 19.00%.

Table 5

Influence of a higher percentage (0.4%) of P-MDI additive in the composition recipe of particleboards with reduced density upon their properties

Raw material recipe (hardwood/softwood)	ρ, kg/m^3	F_v, mg/kg	G_t, %	A_s, mm	f_m, N/mm^2	E_m, N/mm^2	f_{t1}, N/mm^2	SS, N/mm^2
25/75	592.4	512.4	7.9	38.0	11.7	2129	0.27	1.24
30/70	588.5	467.2	6.4	35.2	10.6	2138	0.29	1.05
35/65	594.6	431.9	11.6	50.3	12.3	2282	0.32	1.20

By increasing the percentage of isocyanate additive to 0.4% in the composition recipe of particleboards with reduced density, the following property modifications were achieved compared to the particleboards with 0.25% additive:

- the formaldehyde emission decreased further by 9.99%;
- the swelling in thickness decreased further by 5.95%;
- the surface absorption decreased further by 5.70%;
- bending strength, modulus of elasticity and internal bond did not change significantly anymore;
- the surface soundness increased further by 4.20%.

However, the property improvements are modest. Although the formaldehyde emission registered the lowest values in this variant, the fact that no other significant benefits (especially in bending) are brought by the additional additive amount, the variant with 0.4% additive is considered to be less efficient than the one with 0.25%.

An overview concerning the modification of each property for the recipe 25/75, is presented in Figs. 7-13, comparatively in all four considered variants:

- the dark grey bar = normal density boards, without additive;
- the light-grey bar = boards with reduced density, without additive;
- the orange bar = boards with reduced density, with 0.25% additive;
- the green bar = boards with reduced density, with 0.4% additive,

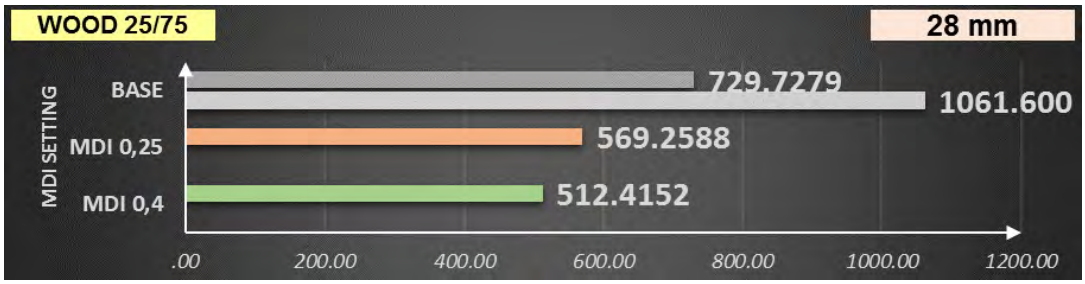

Fig. 7.
Influence of the board density and additive percentage upon the formaldehyde emission (flask method) of the tested prticleboards.

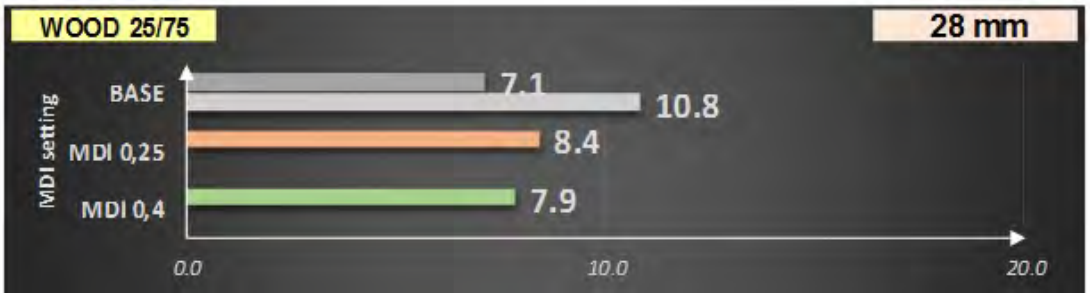

Fig. 8.
Influence of the board density and additive percentage upon the swelling in thickness of the tested particleboards.

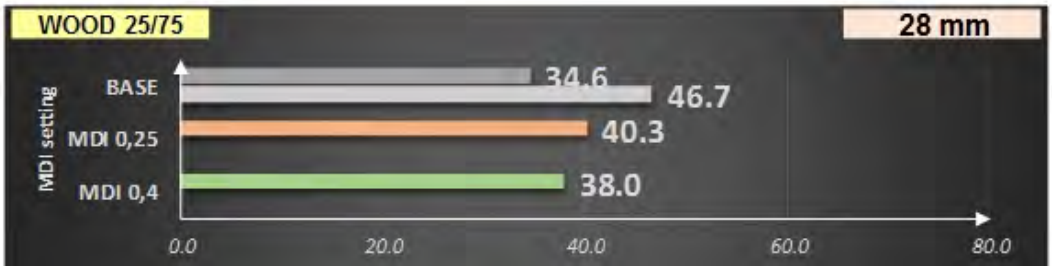

Fig. 9.
Influence of the board density and additive percentage upon the surface absorption of the tested particleboards.

Fig. 10.
Influence of the board density and additive percentage upon the bending strength of the tested particleboards.

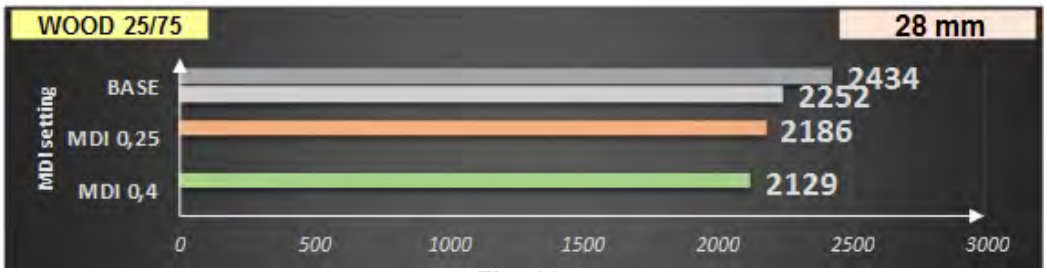

Fig. 11.
Influence of the board density and additive percentage upon the modulus of elasticity of the tested particleboards.

Fig. 12.
Influence of the board density and additive percentage upon the internal bond of the tested particleboards.

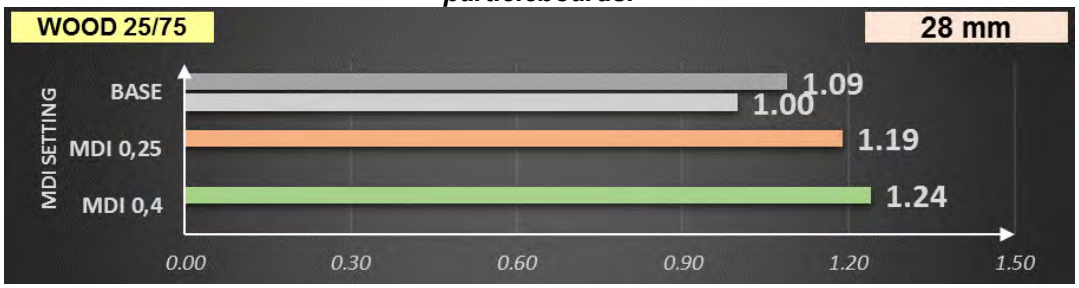

Fig. 13.
Influence of the board density and additive percentage upon the surface soundness of the tested particleboards.

CONCLUSIONS

The conclusions of the present research can be formulated as follows:

1. The percentual participation of hardwood / softwood chips in the composition recipe of normal density 28mm thick particleboards does not significantly influence their formaledhyde emission. Higher percentage of hardwood chips increases density, swelling and surface absorption, without increasing the mechanical performances (bending and tensile strengths) of the boards. Therefore, the best variant among the three studied percentual participations is considered to be 25% (hardwood chips) / 75% (softwood chips).
2. The simple density reduction of particleboards is not a viable alternative because all properties are seriously affected; an additive is compulsorily required.
3. The addition of 0.25% or 0.4% of P-MDI isocyanate additive in the composition recipe of 28mm thick particleboards with reduced density leads to a significant improvement of all properties. The variant with 0.25% P-MDI is considered the optimum one. As compared to the reference variant (normal density boards), it brings:
   - decrease by 4.03% of the density;
   - decrease by 21.98% of the formaldehyde emission;
   - increase by 18.31% of the swelling in thickness;
   - increase by 16.47% of the surface absorption;
   - increase by 10.81% of the bending strength;
   - decrease by 10.19% of the modulus of elasticity in bending;
   - decrease by 25.71% of the internal bond;
   - increase by 9.17% of the surface soundness;
4. The maximum simultaneous reduction of density and formaldehyde emission is possible with 0.4% P-MDI isocynate additive. Compared to the reference variant (normal density boards), this variant allows the:
   - decrease by 4.16% of the density and
   - decrease by 29.78% of the formaldehyde emission.

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EUROPEAN CO-OPERATION IN WOOD RESEARCH FROM NATIVE WOOD TO ENGINEERED MATERIALS: ENGINEERED HYBRID WOOD-BASED PRODUCTS

Peter RADEMACHER, Mátyás BÁDER and Róbert NÉMETH
University of West Hungary, Bajcsy-Zs. u. 4, H-9400 Sopron, Hungary, E-mail: peter.rademacher@nyme.hu, bader.matyas@phd.uni-sopron.hu, nemeth.robert@uni-sopron.hu

Petr KLÍMEK, Václav ŠPRDLIK and Radim ROUSEK, Petr ČERMAK
Mendel University in Brno, Dept. of Wood Science, Zemedelska 3, 61300 Brno, Czech Republic
E-mail: petr.klimek@mendelu.cz, Vaclav.sprdlik@mendelu.cz, radim.rousek@gmail.com, petr.cermak@mendelu.cz

Alexander PFRIEM and Moritz SANNE
Eberswalde University for Sustainable Development, Faculty of Wood Science and Technology, Schicklerstraße 5, 16225 Eberswalde, Germany
E-mail: Alexander.Pfriem@hnee.de, moritz.sanne@hnee.de

Peter MEINLSCHMIDT
Fraunhofer - Institut für Holzforschung - Wilhelm-Klauditz-Institut (WKI)
Bienroder Weg 54E, 38108 Braunschweig, Germany
E-mail: peter.meinlschmidt@wki.fraunhofer.de

Rupert WIMMER
University of Natural Resources and Life Sciences, Institute of Natural Materials Technology
Konrad-Lorenz-Straße 20, 3430 Tulln an der Donau, Austria, E-mail: rupert.wimmer@boku.ac.at

Johann TRISCHLER and Dick SANDBERG
Luleå University of Technology, Wood Science and Engineering
Forskargatan 1, 93187 Skellefteå, Sweden, E-mail: johann.trischler@ltu.se, dick.sandberg@ltu.se

Abstract

Forest-based industries have been continuously developing advanced processes, materials and wood-based solutions, to meet evolving demands and increase competitiveness. Engineered wood products (EWPs) constitute one emerging group of materials aiming at improved property profiles of wood, and provide desired shapes and functionality. In this paper, the main principles for different processes to soften wood and make it more flexible for bending and moulding, such as longitudinal compression, plasticization by water vapour and gaseous ammonia, and a dielectric heating technique, are discussed. Examples of implementation of these techniques for the production of wooden products are presented, and the use of reed canary grass, and a novel technique for embossment of hybrid particleboards are also further discussed.

Key words: *compression; bending; beech; plasticization; thermal-hydro-mechanical processing.*

INTRODUCTION

Only recently has wood been developed to a range of products that are increasingly functional, based on a combination of performance and sustainability requirements. This has been possible because of new industrial processes, which extend not only the size but are also able to modify the properties of natural wood. In addition, manufacturing residues and lower-grade trees can also be used for versatile and more consistent products. The result is a vast array of materials known as engineered wood products (EWPs). Tailoring new bio-based products within the perspective of sustainable development is a philosophy that should be applied to progressively more material-based technologies in different industrial sectors. Ecological concerns are driving the increasing interest in renewable and bio-based resources towards novel products, which can be considered as environmentally-friendly substitutes for petroleum-based products. The advantages of combining renewable sources such as wood with an environmentally acceptable processing technique to achieve new high-quality materials and products, are today more and more recognised.

This paper is based on work by different European research groups in wood science, collaborating in their fields mainly through different COST Actions. Their research give examples of wood processing techniques and material combinations that can be used to manufacture hybrid engineered wood products. The focus has been on methods to improve the possibilities for shaping the wood material and to combine it with other, preferably bio-based, materials.

Wood bending is the general term for a chip-less manufacturing method for shaping wood. In the case of solid wood, the bending is in most cases in fibre direction, but for laminated wood products the bending is both in fibre direction and in transversal direction of the wood.

Embossment is a process where the surface of the wood is partially densified, normally by a steel tool, creating a decorative pattern in the wood surface. This process is comparable with the methods of densification of wood, but the main purpose of the embossment process is not to increase the density of the wood, but to shape the wood surface. Several techniques for wood shaping have been used through a considerable period of time, with classic examples such as the production of nautical vessels, barrels, or sporting goods. Recent developments have shown new possibilities of using these techniques for the development of advanced products.

As for solid wood, new ideas have been focusing on how to increase bendability, i.e. making it possible to bend the wood to small radii and fix shapes when products are in use. Lamination is a well-established technique to build up and optimise construction components of wood of different qualities, or manufacture hybrid engineered wood products. In designing hybrid wood-based materials, the use of bio-based raw materials other than wood, such as straw, are of high interest but can be problematic for both availability and manufacturing reasons. Particleboard has been of special interest to modify because of its widespread use and relatively low raw material costs.

OBJECTIVE

The objective of this paper is to present some examples of recent hybrid wood product development, based on work by different European research groups collaborating in the area of wood science and engineering.

NEW FORMING TECHNIQUES FOR WOOD

The softening or plasticisation of the wood substance is essential for the forming of wood. The shaping of steamed wood has been employed since antiquity and has been carried out in many different ways. Plasticisation of the solid wood with the help of steam or over an open fire in order to make it possible to bend the wood piece without fracture is the most common way of shaping, and is used e.g. for furniture making and ship construction. However, these plasticisation methods are not applicable to all contexts for the intended wood production. Three alternative plasticisation methods will therefore be described here:

- longitudinal compression
- plasticization by water vapour and gaseous ammonia
- dielectric heating technique

Finally, two case studies are presented where these techniques are implemented in new products.

Longitudinal Compression

Compression along the wood fibres makes wood easier to bend to a smaller radius. This is the preferred method to be used for high-density hardwoods. During the modification process, the normally smooth cell walls deform and crinkle and finally look like as shown in Fig. 1. Due to the longitudinal compression, the modulus of elasticity (MOE) and the required bending force decrease dramatically, and ensure a high deformability, much improving the utilization facilities of the wood.

Longitudinal compression requires a high-quality hardwood raw material. Before the compression procedure, the wood has to be plasticized, most practically by steaming. The compression should be 15-25% of the original length. After compression, the degree of compression should remain for 1-2 minutes for relaxation of internal stresses. This relaxation also increases the bendability properties of the wood. The wood can then be bent in a cold state. While the moisture content is high, it is more bendable than in a dry condition, but always easier than uncompressed wood. The main advantage of this method is that the compressed wood can be stored in a cold state for a long time and be bent when it is needed without tedious steaming. This material is primarily used in interior design and for furniture components.

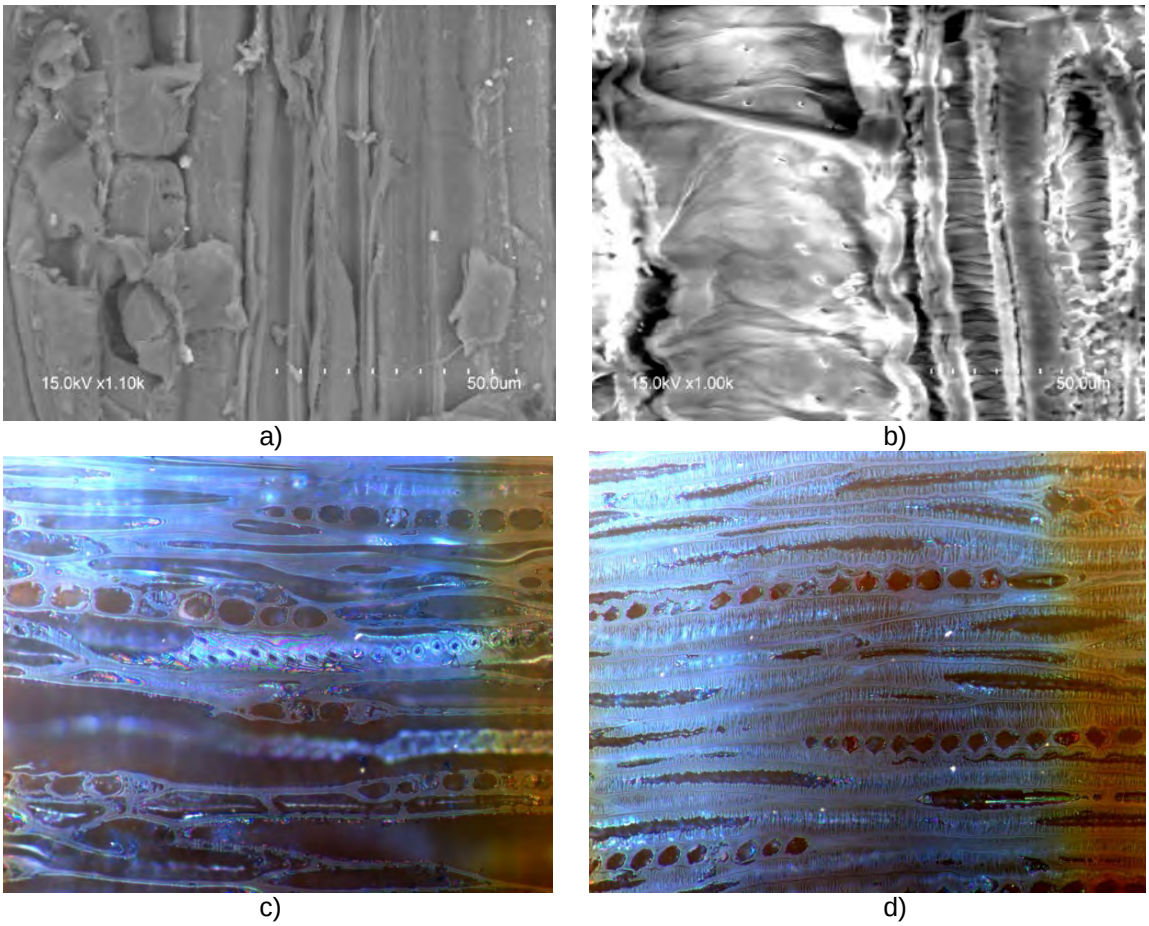

Fig. 1.
Longitudinal compressed oak: radial section a) before, b) after compression, and tangential section c) before, d) after compression.

Plasticization by Water Vapour and Gaseous Ammonia

When wood is plasticized by water vapour, ammonia vapour or by boiling in water, the structure of cellulose and lignin changes and the wood becomes more ductile. Modification by ammonia has been known since the beginning of the 20th century (Kutnar et al. 2015). The most frequently used techniques are boiling in aqueous ammonia solution or in pure ammonia (waterless) and also treatment by gaseous ammonia. Techniques for modification by ammonia have recently received increased interest (Weigl and Müller 2009; Weigl et al. 2009a,b, 2012; Čermák and Dejmal 2013).

Steaming and ammonia treatment are used to change the colour of wood and also to plasticise the wood prior to bending or densification. Mechanisms and processes of the impact of gaseous ammonia treatment – or in combination with steaming – on wood are not very well known. In tests, when wood was treated by water and ammonia vapour, significant changes in the plasticity of the wood have been observed. Šprdlík et al. (2016) tested the mechanical properties for beech modified water and ammonia vapour (Table 1). The result showed that samples which were exposed to water vapour and then to ammonia vapour obtained increased flexibility, i.e. exhibited low MOE and modulus of rupture (MOR) values, Fig. 2.

Table 1

Conditions of the treatments and average density per series. R – untreated reference, and samples treated with W – water vapour, A – ammonia vapour, W+A – water and ammonia vapour

Type of treatment	*No. of samples*	*Density [kg/m^3]*	*Time of treatment [min]*	*Temperature [°C]*
R	***15***	***640±17***	***-***	***-***
W	***15***	***647±23***	***12***	***100***
A	***15***	***633±20***	***180***	***up to 60***
W+A	***15***	***628±26***	***12+180***	***100 + up to 60***

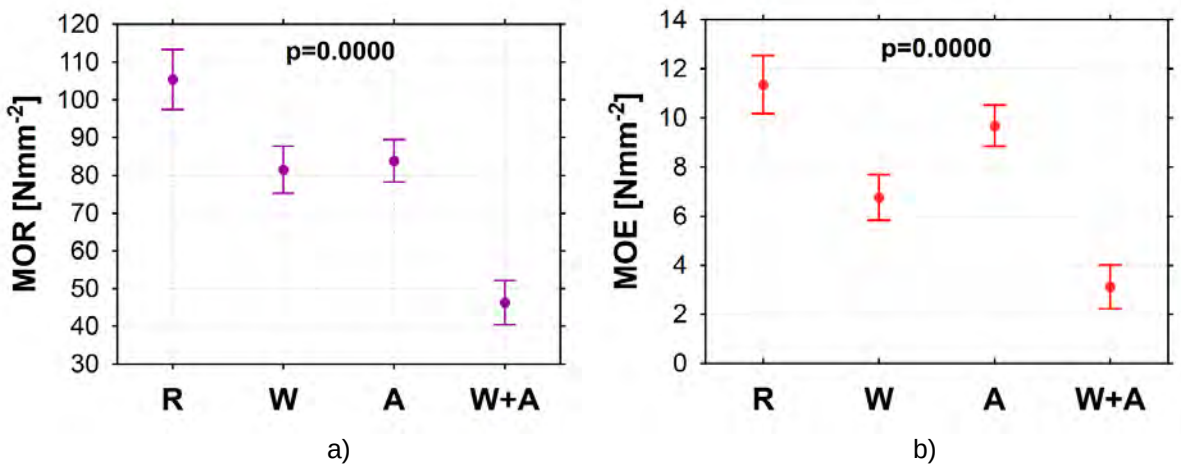

Fig. 2.
Effect of ammonia treatment on properties of beech: a) modulus of rupture (MOR) and b) modulus of elasticity (MOE). R – untreated reference, and samples treated with W – water vapour, A – ammonia vapour, W+A – water and ammonia vapour.

The load-displacement curves in Fig. 3 exemplify the considerably increased flexibility of samples modified by water and ammonia vapour. The untreated reference samples exhibit a significant part of the elastic deformation and failure of these samples occurred immediately after transition to the viscoelastic and plastic deformation part of the deformation curve. Modification with water vapour or gaseous ammonia results in a higher proportion of plastic deformation until failure, and failure occurs gradually. Samples modified with a combination of water vapour and gaseous ammonia result in a very high proportion of viscoelastic and plastic deformation, and the failure of the samples did not occur completely, even when the strain was 100% higher than in the case of the other modification treatments.

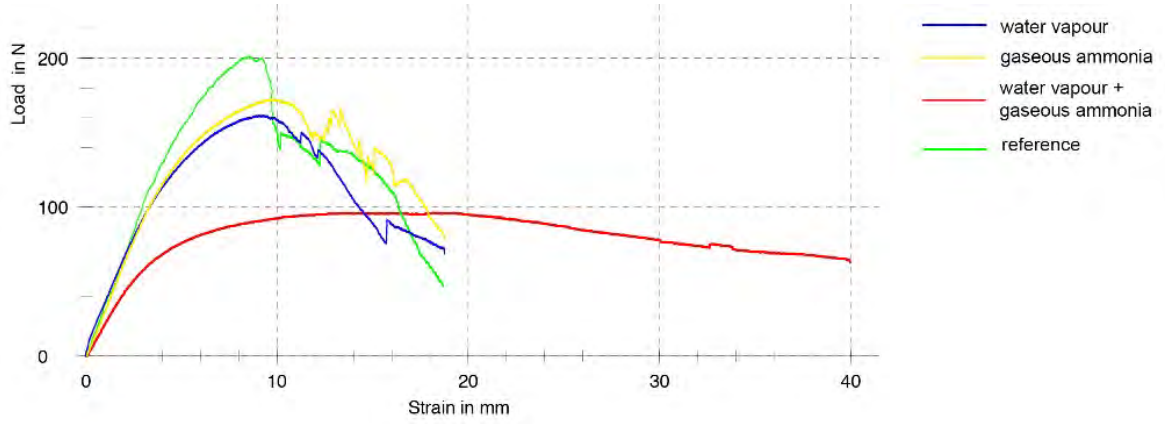

Fig. 3.
Load-displacement curves for treated and reference samples.

Dielectric Heating of Solid Wood

A dielectric heating technique for solid wood bending — i.e. heating, plasticizing and drying the wood to be bent in one step — is a highly productive technique. The main purpose for using dielectric heating is to decrease the time of the bending process. In practise, this means reducing the time to bend and dry a straight piece of solid wood from a moisture content of about 25% to 6-8% from about 3 days to just 10 minutes. The use of dielectric heating for plasticizing wood for shaping it has been studied for several years (Sandberg et al. 2013). The theoretical approach is well documented by Torgovnikov (1993), and Navi and Sandberg (2012) have in their work thoroughly described a process for bending solid wood by using dielectric heating.

The short processing cycles give high demands on controlling the moisture content, temperature and the strain fields that occur in the wood during processing. Fig. 4 shows the temperature and moisture content in beech during a bending process (mean values for 21 wood

pieces). The temperature was measured at the centre of each piece. The same figure also shows the rejection level of curved pieces, i.e. curved pieces that do not satisfy the requirements in bending radius or are not free from damage, cracks etc. The graph shows the rejected proportion as a function of time. During the heating stage, the rejection is by definition 100% as the pieces are not yet curved. During the bending stage, the temperature is almost constant and the moisture content drops rapidly. At the end of this stage, the moisture content is about 10%. If the process was stopped at this time, the rejection proportion would be 100% as a consequence of spring-back of the curved pieces.

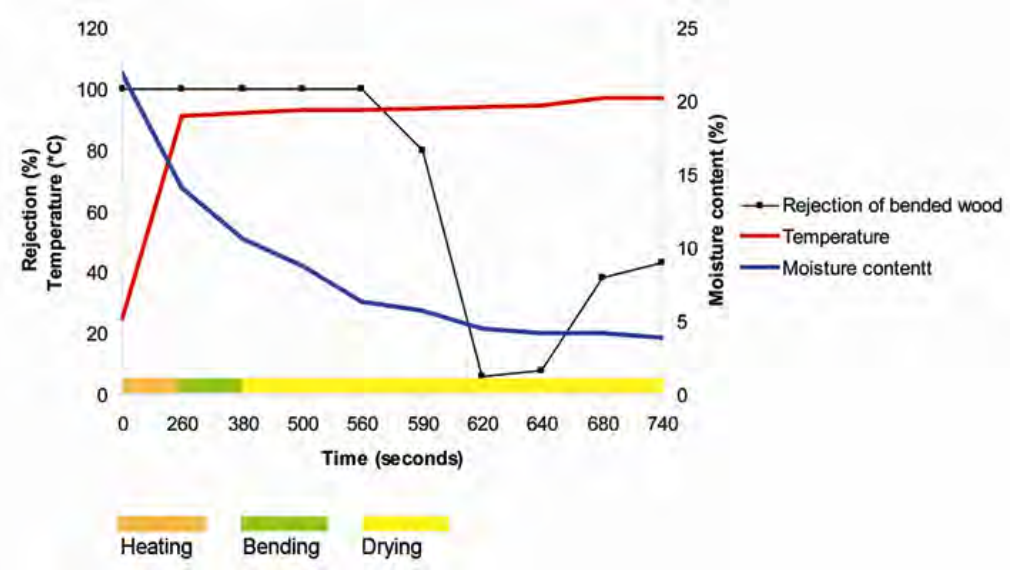

Fig. 4.
Temperature, moisture content and rejection as a function of time during the bending of solid beech wood using dielectric heating.

During the drying stage, the moisture content is lowered to a level where the curved shape is fixed at the required radius. In Fig. 4, it is clear that the moisture content falls as long as the drying continues until no water – or a low amount of water – remains in the wood. The temperature rises from 95°C to about 98°C during 360 seconds. The rejection falls from 100% to about 5% during a time period of about 60 seconds. The process should be stopped at this time period, to obtain a low rejection of wood pieces. If the drying continues, the rejection level of bent pieces increases again, and when the bending process is stopped, as in the example in Fig. 4, the rejection rate is as high as 40%. The curves in Fig. 4 show that there is a relatively short time span during which the process should be stopped to ensure a low rejection rate of the bent pieces while the moisture content is not too low. The optimal processing time depends of course on the energy input into the wood.

Different types of damage that may occur in the wood during the bending process are related to the rupture of cell walls if the steam generated within the cells of the wood finds difficulty in escaping, flashover in the dielectric field, tensile or compressive rupture related to incomplete softening of the wood or simply a problem with the structure of the wood (deviating fibre orientation, knots etc.).

Case Study 1: Design of a Stool

The ammonia treatment technique has been tested in a case study where a stool with wood details of extreme radii was designed and produced. The stool itself is designed as a spring, which gives the user the option to change position and allows the sitting person to swing. This feeling is relaxing and comparable to the movement of a child's cradle. Due to the construction, concerns about mechanical stability of the stool appeared, and a numerical analysis was proposed to ensure safety by assessing the maximal possible loading relative to the strength of the wood. A laminated structure was used because of the composite behaviour of the material.

Two types of experimental stools were designed to show the potential of veneer treated with ammonia. These geometries were analysed by using the finite element method (FEM). Numerical simulation of static structural analysis was done for the loaded stool. The laminate was defined as a linear-elastic orthotropic material with seven layers. Boundary conditions agree with standard furniture testing. A finite element model of one geometry is shown in Fig. 5.

The resulting values of stress in the material are approximate. However, Figs. 5a-b shows stress distribution in direction Y (perpendicular to the plane of the seat) in the modelled chairs. The red colour indicates areas with the maximum tensile stress and the blue colour indicates areas with the maximum compressive stress. Chair type B has a larger tensile area than chair type A, but for chair type B the value of this stress is about 24% lower (3.5 MPa) than for chair type A (4.6 MPa).

The equivalent stress distribution in Fig. 5c-d shows similar results as in Figs. 5a-b, but this time for all three directions X, Y and Z. The blue colour indicates the minimum equivalent stress and the red colour indicates the maximum equivalent stress in chairs. The approximate maximum value of equivalent stress is 0.4 MPa for chair type B and 0.53 MPa for chair type A. As the results show, the area of maximum equivalent stress is very small for chair type A compared with chair type B, but, as the results show, about 25% higher.

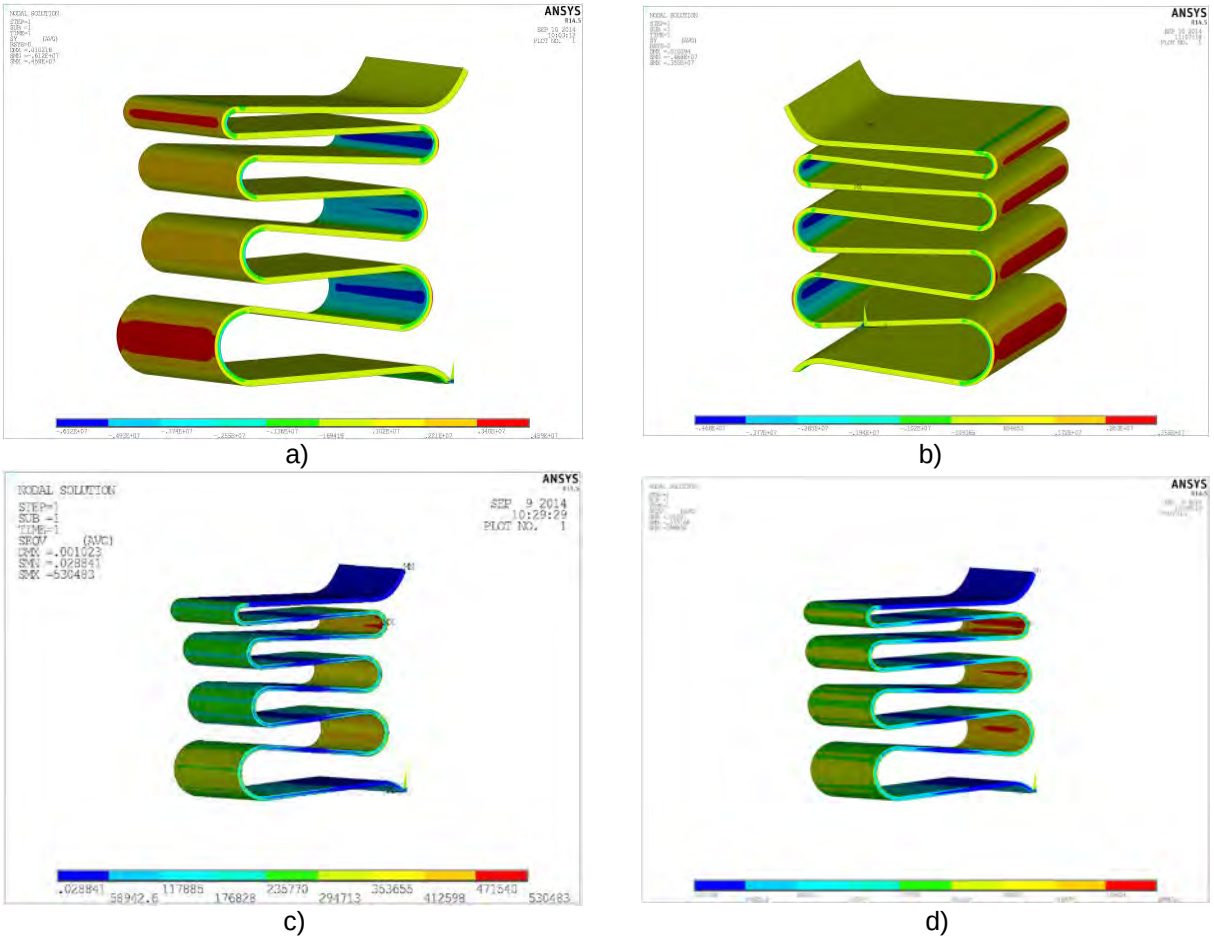

Fig. 5.
FEM model of a stool of ammonia treated beech veneer: stress distribution in Y-direction for chair a) type A, b) type B, and equivalent stress distribution in individual chairs for chair c) type A, d) type B.

Case Study 2: The Wooden E-Bike

Ash is a strong, durable and therefore widely used wood for parts and components where dynamic forces are dominant. The Wooden E-Bike is an example of such a dynamically stressed structure. There are bending and torsion forces in a bike frame. Due to the design, the Wooden E-Bike could not be manufactured without the use of engineered wood (Fig. 6).

Fig. 6.
Selected manufacture steps of the Wooden E-Bike: lamination of 5 mm thick ash boards to a "glulam" component (upper), CNC milled bicycle components (middle), and the finished E-Bike in use.

Ash boards with a thickness of 5 mm were used to achieve glued laminated (glulam) components, and were bent in form to achieve a straight grain along the main stress direction. In this way, small failures in the wood material were "equalized" and the risk of failure in the finished bike frame was reduced to a minimum. The moulded components were then machined with the help of a 3-axis computer numerical control (CNC) machine. The lightweight frame construction with its thin walls and large squared profile has a capacity comparable to standard aluminium bike frames. Except for the motor, all electrical parts, Bowden cables and hydraulic tubes are hidden inside the hollow bike frame and thereby protected.

Different types of adhesives were tested during the project. The choice of adhesive influences the stiffness of the frame structure which is also influenced by the amount of adhesive applied on the

lamellae and their open time during gluing. For example, a reactive polyurethane (PUR) adhesive gave a more flexible construction when the open time was as long as specification (max. 30 minutes), rather than having pressure applied immediately after application of the adhesive. Another feature with different adhesives is the adjustable moisture content by choosing either a water-based or an isocyanate-based adhesive. In the bending process, the lamellae have to be slightly watered in order to make the surface more flexible. This water helps to extend the open time when using water-based adhesives and enhances the quality of the PUR glue-line.

PARTICLEBOARD DEVELOPMENTS

The main motivation for wood-based composites has been the compensation for the heterogeneity and anisotropy of solid wood, large-panel dimensions, a good achievable surface quality, and low-cost production and thereby a cheap product compared to high-quality sawn timber. Forecasts show that by 2020 the European consumption of wood and wood fibre raw material could be as large as Europe's combined forest growth increment. An increasing proportion of the forest raw material is expected to be used as fuel for heating, as propellant fuel or to generate electricity. This means an increasingly tight competition for wood between the board industry and the energy-conversion industry and a need for the board industry to find new raw material sources.

Particleboard is an important base for furniture production and interior use, and the production process is in general optimized for wood as the main raw material. The cost pressure and capacity utilization in the particleboard industry are leading to a demand for large amounts of cheap wood as raw material, and further product development. Examples of how to substitute wood in particleboard production and to develop the function of the board is presented below.

Reed Canary Grass in Hybrid Particleboards

High productivity in the boreal regions makes reed canary grass (*Phalaris arundinacea* L.) interesting as a raw material for several applications where wood is today the main raw material. One possible application is in board manufacture, e.g. as a substitute for wood in the core of multi-layer particleboards. The properties of reed canary grass must, however be modified to meet the industrial standards for particleboard production and for the mechanical properties of the boards. Alternatively, different adhesives can be chosen.

Lignocellulosic raw material is a resource continuously provided by nature and encompasses a variety of specifications. Within the scientific classification, the plants producing lignocellulosic raw material relevant to particleboard production are gymnosperms (conifers), and the angiosperms, also known as "flowering plants", to which belong among others the grasses and the broad-leaved trees. Table 2 presents a partition of various species producing lignocellulosic raw material which is or can be interesting for the particleboard producing industry due to their high annual production rates.

Table 2

Chemical composition as a percentage of the dry mass of selected species (Trischler et al. 2013) C – celluloses, L – lignin, HC – hemicelluloses, A – ash content

Species	C	L	HC	A	References
Wheat straw (*Triticum aestivum* L.)	38-41	8-15	29-31	6.0-6.3	(Bridgeman et al. 2008; Hartmann et al. 2009)
Miscanthus (Miscanthus x giganteus)	43-49	11-19	29-30	2.6-6.0	(Hartmann et al. 2009; Hodgson et al. 2011)
Reed canary grass (*Phalaris arundinacea* L.)	30-43	8-11	25-30	1.3 -6.0	(Dien et al. 2006; Bridgeman et al. 2008; Jansone et al. 2012)
Cup plant (*Silphium perfoliatum* L.)	36	12	18	9	(Wulfes 2012)
Softwoods	40-45	25-35	25-30	0.2-0.4	(Fengel and Grosser 1975) (Pettersen 1984)
Hardwoods	40-50	20-25	25-35	0.2-0.8	

Reed canary grass (RCG), which might be a potential source of lignocellulosic raw material suitable for particleboard production, was used to produce particleboards. MUF (melamine urea formaldehyde) and wheat protein (vital gluten) were used as adhesives to produce particleboard with RCG either alone or in combination with wood, Fig. 7.

Fig. 7.
Design of the particle boards: a) a surface view of the core layer consisting of pure RCG chips, b) a cross-section view of a three-layer board showing the extent of substitution, and c) surface and dimension of a three-layer board with an RCG core layer (Trischler et al. 2013)

It was possible to produce particleboard of RCG (Fig. 7), but none of the boards passed the requirements of the relevant standards. However, the tests were helpful for the evaluation of the production parameters and properties of particleboard containing large amounts of lignocellulosic raw materials of non-wood plants such as RCG. The particles were used in a natural manner to obtain a reference value without any pre-treatment and because any additional processing of the raw material would lead to increased production costs which are to be avoided. The results showed that it is essential to find an efficient technique for surface treatment of the raw materials from non-wood plants if they are to be used in combination with wood in conventional particleboard production. Unfortunately, such pre-treatments are cost- and time-consuming and, as a result, all the cost advantages of these non-wooden lignocellulosic raw materials such as agricultural residues are lost. There is a further logistical problem which the lignocellulosic raw materials have in common: Agricultural residues, in particular, are often available in large amounts only once or at most twice a year and have to be stored over the rest of the year without losing their mechanical properties. It was therefore necessary to find a way to overcome both the surface treatment and the logistical and storage problem.

Creating 3D Surfaces on Particleboard by Pattern Imprintment

The objective of this work is the development and application of imprinted patterns on both sides of a particleboard, with the purpose of locally increasing the surface layer density without changing the given board thickness. This imprintment should be done as a post-treatment, which means it is applied as a final processing step on finished boards. It is hypothesized that imprinted particleboard will still demonstrate some mechanical integrity. The purpose of this treatment is to create three-dimensional (3D) surfaces as a new design feature. In addition to laboratory tests the obtained density changes are assessed by the finite element modelling.

Test samples were obtained from single-layer commercial particleboard. Boards with three different thicknesses (12, 15 and 18 mm) were tested. Sample length was set as twenty times the thickness plus 50 millimeters. From each single board 50 samples were cut, 25 for the embossment experiments, and 25 reference samples.

Samples were impressed in a 60-ton hydraulic press. For all prepared samples, only a single pattern was employed, which was manufactured and machined from a 3 mm thick stainless steel plate (Fig. 8). Patterns were impressed on both sides of the boards, with the top side pattern shifted by a half hexagon diameter in longitudinal direction to the bottom side pattern, to prevent superposition. For the first 10 impressment tests, the press pressures was varied between 0.7 to 1.2 MPa, until the optimal press pressure for full-pattern impressment was achieved (found at 0.8 MPa). Some boards were also preheated to 150°C, to achieve a thermal softening effect of the board surfaces. The thermal treatment process was set to 15 minutes, with a subsequent pressing time of 10 minutes.

Fig. 8.
Particleboard with imprintment pattern plates on both sides of the panel.

The finished panels had a clearly visible pattern (Fig. 9). Due to the impressments, the bending data have significantly declined. The 12 mm thick board shows the MOR being decreased by 71% and MOE down by 80%. The other board thicknesses were likewise. It is clear that a post-production impressment has destructive consequences on the board. During impressment the cured resin, which is in part crystalline and therefore brittle, will be locally distorted with the consequence the adhesion will be in part lost. The glued wood particles also got broken and separated, which also contributes to the loss of internal integrity.

Fig. 9.
Surface coating of imprinted pattern particleboard.

To evaluate the effects of imprinted surface patterns on particleboard properties a finite element model (FEM) was employed. The main focus of this approach was the assessment of different imprinting shapes and sizes, and their effects on local surface densification. Several models with the pattern diameters 20, 25 and 35 mm were applied, with the geometric shape kept constant (Fig. 10).

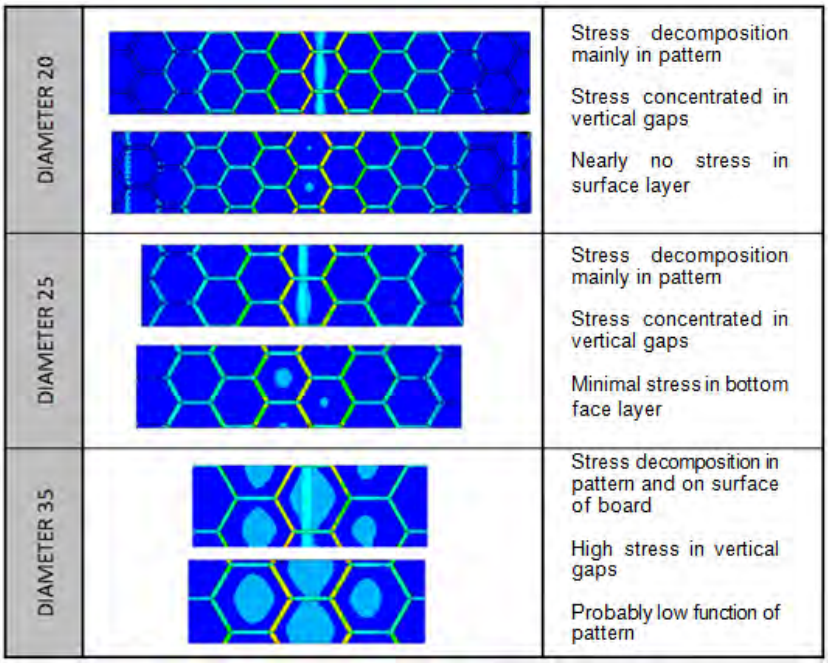

Fig. 10.
FEM results for the imprinted density patterns, computed with different shape sizes.

The finite element modelling has shown that all employed patterns have the potential of also creating mechanical support. The first and second hexagons' diameters (20 mm and 25 mm) are nearly the same, with some low stresses in the top face layers while loading. The third pattern (35 mm) was found ineffective and was therefore excluded (Fig. 10).
Even with severe loss of the bending performance, the imprinting pattern treatment into particleboard can be seen as successful. By imprinting a hexagonal pattern, the surface has been significantly altered, while at the same time keeping a minimum of internal mechanical integrity. This type of board will not be used for high-strength purposes; this new type of three-dimensional surface is seen as a new figuration or design. With proper coating treatments, applications can be found such as for wall

cladding, ceiling cladding or for decorative products. With the shown imprinting treatment, a new type of 3D-surface particleboard was created, which may find some new market niches.

CONCLUSIONS

Research on engineered wood products is intensive, with several research groups in Europe trying to understand wood-shaping processes better, and to develop new improved hybrid engineered products that are produced in an environmentally friendly way. The main challenges for the future are finding economical and environmentally friendly production methods and scaling up to profitable industrial applications.

Several promising techniques for increasing the formability of wood or wood composites are available, and methods such as longitudinal compression, plasticization by water vapour and gaseous ammonia or by dielectric heating have been presented here.

Examples of how to substitute wood in particleboard production indicate that by using particles of non-wood species such as straw or grass in their natural way leads to boards with a low mechanical performance due to bonding problems when conventional adhesives are used. Therefore, a pre-treatment process has to be developed which leads to better usability of the monocotyledon particles and which can easily be integrated into the conventional particleboard production process. Studies into the effect of imprinted density patterns in the surfaces of particleboard have revealed that bending properties (MOE and MOR) are significantly lowered. However, imprinting a predefined shape onto surfaces of particleboards was successful, and with the created 3D surface new applications and product opportunities are created for particleboard.

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SOME PROPERTIES OF DECORATIVE MDF PANELS MANUFACTURED FROM COLOURED WOOD FIBERS

Derya USTAÖMER
Karadeniz Technical University, Faculty of Forestry, Forest Industry Engineering
61080, Trabzon,Turkey
E-mail: uderya@ktu.edu.tr

Ayfer DÖNMEZ ÇAVDAR
Karadeniz Technical University,Faculty of Architecture, Interior Architecture
61080, Trabzon,Turkey
E-mail: donmez@ktu.edu.tr

Abstract

In this study, some properties of Medium Density Fiberboard(MDF) panels manufactured from the coloured fibers with various dyes were investigated. The effects of these dyes on the colour change, surface roughness parameters, water absorption and thickness swelling of the MDF panels were evaluated. The results showed that while the highest total colour change value was obtained with the MDF samples manufactured from the coloured fibers using methylene blue, the lowest total colour change value was recorded with the MDF samples manufactured from the coloured fibers using indigo carmine. The surface roughness values of the coloured MDF samples were found lower than that of the control MDF sample.The water absorption(WA) and thickness swelling(TS) increased in the coloured MDF samples. It was found that, all values changed depending on the type of dyes.

Key words*: MDF; Coloured MDF; fiber; dye; colour change; surface properties; physical properties.*

INTRODUCTION

Nowadays, architects, interior designers and furniture manufacturers have been in search of high-quality wood based panels which are creative and ambitious solutions for furniture and room design to allow for the highest level of customer satisfaction. The panel producers have developed new technologies and products to satisfy the customer's needs.

Wood based panels are widely used in furniture manufacturing, strengthened floor, wall panels for interior decoration. Due to the finer texture of the fibers used in manufacturing Medium Density Fiberboard (MDF) it is smoother than other wood based panels. The uniform texture and density of the fibers create a homogenous panel that is very useful as a core for paint, thin overlay materials, veneers and decorative laminates (Maloney 1993). MDF is among the most stable of the mat formed panel products also, it can be used replacing a lot of natural wood and have significant economic benefits.

Coloured MDF provides many facilities to designers inspiring their designs. It can be much more than skin deep among decorative panel products. The through-dying process used in its production means that coloured MDF is perfect for a wide variety of applications (URL 1; URL 2).

Dyes and pigments are most important colouring agents. They are extensively used for many areas such as textile, food, cosmetics, plastics, pharmaceutical, ink, paint and paper industries (Gürses et.al 2016). Dyes may be classified according to several ways such as appplication class, chemical structure, and end use (Adegoke and Bello 2015). Safranin is among the oldest known synthetic dyes. It is primarily used as food dye. Safranin is also used as dye for cotton, wool, bast fibers, silk, leather, tannin, paper (Guler et al. 2016) and plant microscopy (Bond et al. 2008). Methylene blue is widely used for dyeing silk, cotton and wood. It is known that it has strong adsorption onto solids (Shahryari et al. 2010). Indigo carmine is one of the most useful dyes that is used as colourant in the textile industry, and additive in pharmaceutical tablets and capsules (de Carvalho et al. 2011). It is also used as colourants for food (Fleischmann et al. 2015).

Many studies have been conducted to investigate the effect of various dyes for wood and wood materials (Hu et a 2016; Özen et al. 2014; Yeniocak et al. 2015; Göktas and Yeniocak 2016).

OBJECTIVE

The objective of this study was to evaluate the colour change, surface roughness parameters, water absorption and thickness swelling of the MDF panels manufactured from the coloured fibers with various dyes (indigo carmine, methylene blue and safranin) which they are commonly used in different areas.

MATERIALS AND METHODS

In this study, commercial softwood fibers were used as raw material. As dyes for colouration of fibers, indigo carmine, methylene blue and safranin powders were used. The dyes were supplied by Merck Co., Germany.

Colouration of Fibers

Aqueous solutions were prepared at 0.1 % concentration of each dye for 2L water. Wood fibers were treated with the solutions of indigo carmine, methylene blue, and safranin by soaking in a tank for 24 hours. Later fully saturated coloured fibers were dried in a laboratory oven until they reach 2–3% moisture contents. Dried fibers were also screened to separate fiber bundles. The coloured fiber samples were represented in Fig.1.

(a) (b) (c) (d)

Fig.1.
(a): fibers without dye(uncoloured), (b): coloured fibers with safranin, (c): coloured fibers with methylene blue, (d): coloured fibers with indigo carmine.

Manufacture of MDF Panels

The fibers were mixed in a rotary drum equipped with resin spraying unit. Urea formaldehyde (UF) was used as adhesive. The mats manually were formed and pressed in a press for 170 °C, 7 min. After manufacturing, the MDF samples were conditioned in a climate room with a temperature of 20 °C and a relative humidity of 65 °C until they reach to equilibrium moisture content before tests. Fig.2 shows the coloured panel mats.

Fig. 2.
Coloured panel mats.

Colour Measurements

The colour measurements of the coloured samples were carried out using by Mitech Technology MCD-100 spectrophotometer according to the CIE *L*a*b** system (HunterLab 2008). The Δ*L**, Δ*a**, Δ*b** colour coordinates, and total colour change (ΔE*) occurring in the samples were calculated.

Surface Roughness

The surface roughness parameters such as *R*a, *Rq* and *R*z of the MDF samples were measured using Mitutoya Surftest SJ-210 instrument according to DIN 4768 standard.

Water Absorption and Thickness Swelling

The water absorption and thickness swelling values of the MDF samples for 24 h were determined according to EN 317 standard.

RESULTS AND DISCUSSION

Colour measurements

Some images of the uncoloured/coloured samples were given in Fig.3. As can be seen in Fig.3, the dyes gave different colours to fibers. The colour of fibers turned into red with safranin dye, to blue with methylene blue, and to green colour with indigo carmine.

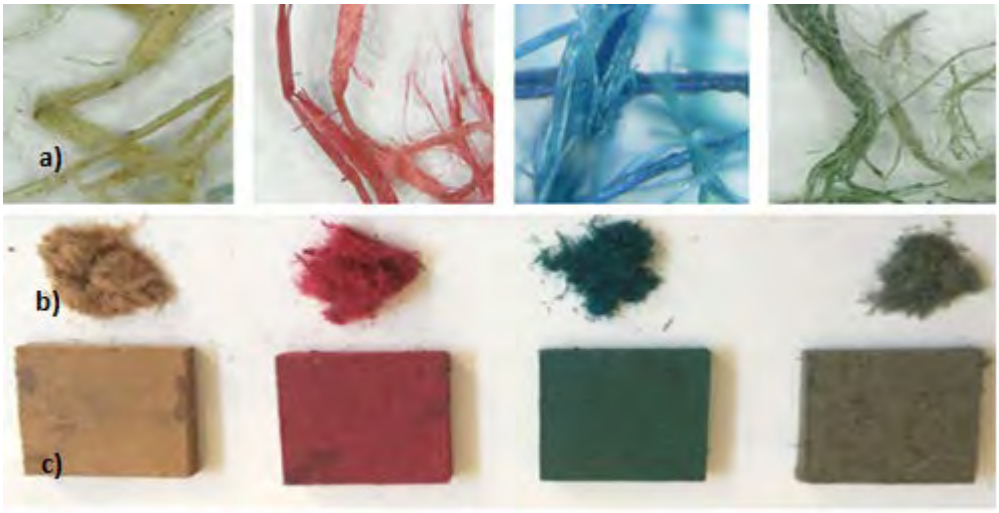

Fig.3.
a) Microscopic images of the fibers b) uncoloured/coloured fibers
c) uncoloured/coloured MDF samples.

The colour measurement results of the coloured fibers and coloured MDF panels were represented in Table 1.

Table 1

Colour changes of fibers and MDF samples

	Groups	Δ*E**	Δ*L**	Δ*a**	Δ*b**
MDF	Red MDF	27,8	-14,8	18,2	-149
	Blue MDF	34,4	-14,4	-22,1	-221
	Green MDF	17,4	-5,1	-10,7	-127
Fibers	Red fiber	8,3	-4,4	7,0	2
	Blue fiber	12,4	-7,5	-5,3	-83
	Green fiber	5,7	-1,8	-3,9	-37

*The MDF panels with safranin labeled as red MDF; the MDF panels with indigo carmine labeled as green MDF; the MDF panels with methylene blue labeled as blue MDF.
** Control groups were taken as reference for color measurements.

Table 1 displays the colour change values of the coloured MDF panels. Each groups showed different color change depending on the type of dye. The ΔE^* value was highest for blue MDF samples manufactured from the coloured fibers with methylene blue. This value was found to be 34,4. The lowest ΔE^* value was found to be 17,4 for green MDF samples manufactured from the coloured fibers with indigo carmine. The ΔL^* was determined to be the lowest value (-5,1) for green MDF samples. The negative ΔL^* values were found similar for red MDF and blue MDF samples.

The results also show that the chromaticity coordinates, the Δa^* and Δb^* for the MDF samples were found as positive and negative values. Positive and negative values of Δa^* indicate a tendency of sample surface to become reddish and greenish. Positive and negative values of Δb^* indicate a tendency of sample surface to become yellowish and bluish, respectively (HunterLab 2008). As can be seen from Table 1, the highest Δa^* value was 18,2 for red MDF samples. This positive value represents a tendency to red colour. The negative Δa^* values of green MDF and blue MDF indicate

that they have tendency to green colour. All Δ*b** values of the MDF samples were found as negative. The highest negative Δ*b** value was obtained from blue MDF samples.

Table 1 also displays that the same trend was observed for colour change values of the coloured fiber samples. The highest Δ*E** value was found to be 12,4% for blue fibers, the lowest Δ*E** value was found to be 5,7% for green fibers. The lowest Δ*L** value was recorded from green fibers. The highest Δ*L** value was found for blue fibers. Additionally, it is seen that all colour change values of the MDF panels were found higher than those of the fibers. Probably, these differences can be ascribed to panel manufacturing parameters, additives, and etc.

Surface properties

The surface parameters values of the control MDF and coloured MDF panels manufactured with different dyes were represented in Table 2. The evaluation profiles of the MDF samples also were given in Fig.4-7.

Table 2

Surface roughness values of MDF samples

Groups	Ra(µm)	Rq(µm)	Rz(µm)
Red MDF	4,85	6,27	31,96
Blue MDF	5,02	6,26	29,75
Green MDF	5,35	6,64	31,04
Control	5,68	7,11	32,59

As can be seen from Table 2, the surface roughness of all coloured MDF samples decreased in comparison with the control MDF sample.The highest surface parameters (Ra, Rq, Rz) were obtained from the control MDF samples. The red MDF samples resulted in the smoothest surface with the Ra value of 4,85 µm, while corresponding value for the control sample was 5,68 µm. The lowest value of Rq was found to be 6,26 µm for blue MDF samples; the highest value of Rq was 7,11 µm for control MDF samples. While the highest value of Rz was recorded from the control MDF samples, the lowest value of Rz was found for blue MDF samples. The values changed depending on the type of dyes. It is known that the degree of surface roughness is a function of manufacturing parameters and raw material properties (Hiziroglu and Kosonkorn 2006).

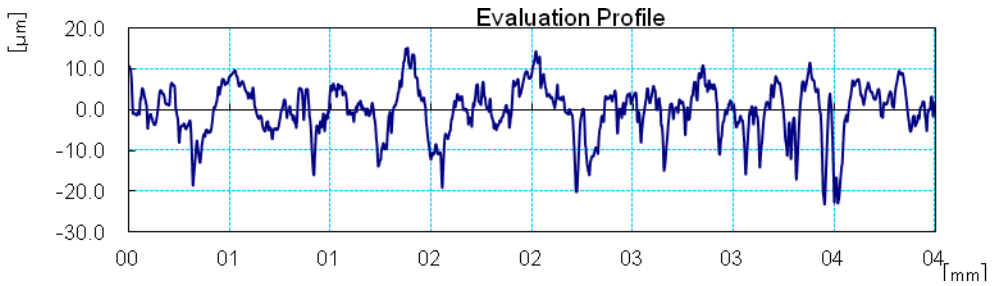

Fig.4.
Evaluation profile of red MDF.

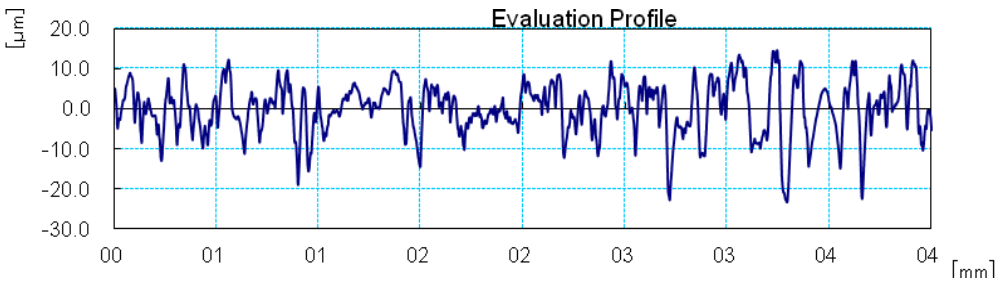

Fig. 5.
Evaluation profile of blue MDF.

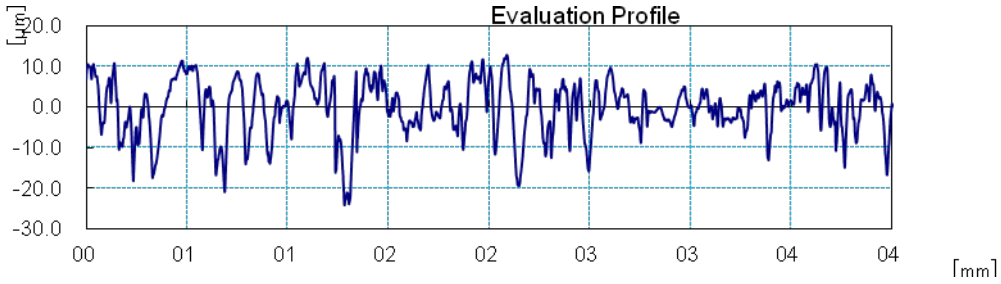

Fig.6.
Evaluation profile of green MDF.

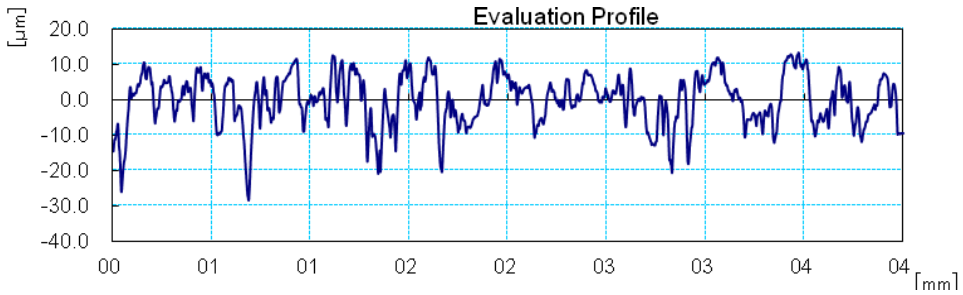

Fig.7
Evaluation profile of control MDF.

Water Absorption and Thickness Swelling

The results of the water absorption and thickness swelling of the MDF panels were given in Table 3.

Table 3

Water absorption and thickness swelling of MDF samples

Groups	WA(%)	TS(%)
Red MDF	52,48	20,60
Blue MDF	54,87	21,82
Green MDF	60,66	23,12
Control	32,03	14,80

As can be seen in Table 3, the water absorption values of the coloured MDF samples for 24h were found higher than the value of control MDF sample. The WA value was found to be 32,03% for the control MDF panels; the highest value was obtained to be 60,66% for green MDF panels. The lower value was found for red MDF panels among the all coloured MDF samples. Similarly, same trend was obtained for TS values. The lowest TS value was found to be 14,80% for the control MDF panels. The highest TS value was determined to be 23,12% for green MDF panels, respectively. The results demonstrated that the type of dye in the MDF panels caused an increase on the water absorption and thickness swelling values. This increase can be attributed to chemical structure of these dyes.

CONCLUSIONS

The findings of this study showed that the dyes have effect on the some properties of the MDF panels such as surface roughness, colour change, water absorption and thickness swelling. All values showed different trend depending on the type of dye. The highest color change values were obtained from blue MDF panels. The highest surface roughness parameters were recorded from the control MDF samples. The water absorption and thickness swelling values of the coloured MDF panel samples increased depending on the type of dye. It appears that the alternative dyes, pigments and additivities can be used to improve the some properties of panels.

As a conclusion, coloured MDF panels can be used effectively without laminating or edge banding in many areas where they have potential usage.

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SOME PROPERTIES OF FIBERBOARDS MANUFACTURED WITH ASH (*Fraxinus excelsior* L.) SAWDUST

Derya USTAÖMER
Karadeniz Technical University, Faculty of Forestry, Forest Industry Engineering
61080, Trabzon, Turkey
E-mail: uderya@ktu.edu.tr

Elif TOPALOGLU
Giresun University, Technical Sciences Vocational School, Architecture and Urban Planning Department
28000, Giresun, Turkey
E-mail: elif.topaloglu@giresun.edu.tr

Nurgül AY
Karadeniz Technical University, Faculty of Forestry, Forest Industry Engineering
61080, Trabzon, Turkey
E-mail: nay@ktu.edu.tr

Abstract

The objective of this study was to evaluate the effect of ash (Fraxinus excelsior L.) sawdust on the some properties of Medium Density Fiberboard (MDF). The MDF panels were manufactured from pine fibers and ash sawdusts at the different ratios (10%, 15%, and 20%). Some properties of MDF panels such as surface roughness parameters (R_a, R_z), colour change (ΔE^*), water absorption (WA), thickness swelling (TS) were determined depending on the ratio of ash sawdust. As a result, it was found that the surface roughness parameters and colour change values increased with increasing the ratio of sawdust, the highest surface roughness and colour change values were obtained from the panel type C. However, the water absorption and thickness swelling values decreased with increasing the ratio of sawdust. The lowest values were obtained from the panel type C.

Key words: *fiberboard; ash sawdust; physical properties; surface roughness; colour change.*

INTRODUCTION

In the recent years, there has been a remarkable growth in the wood based panel industry in the world. This rapid industrial growth has increased the demand of wood raw materials for the manufacturing process. One of the main problems for panel manufacturers is a decrease in the resources of wood raw materials. This problem has pushed the manufacturers to find alternative solutions.

Medium density fiberboard (MDF) industry, as well as other industries, is negatively affected by a decrease in the wood raw materials (Akgül and Tozluoğlu 2008). A solution to this problem could be to find alternative raw materials or to use wood resources such as harvesting residues, annual plants, agricultural wastes, bark, furniture and lumber plant wastes, recycled paper, etc. (Akgül and Çamlibel 2008).

Shavings and sawdust have various uses such as fuel uses, chemical uses, fiber and wood-based board uses (Harkin 1969). Many studies have been carried out related to the use of some wastes and various forest residues in the manufacture of wood based panels (Chow 1979; Nemli and Aydın 2007; Akgül and Tozluoğlu 2008; Ayrilmis *et al.* 2009; Bardak *et al*. 2010; Yel *et al*. 2014).

The using areas of wood based panels such as fiberboard and particleboard are the manufacture molding, laminated flooring, overlaid panels for cabinet and furniture industry. When these panels are preferred to use as substrate for thin overlays, their surface properties have importance for quality of final products (Hiziroglu and Suzuki 2007)**.** The type of raw material, the content of resin, pressing, the size of particle, moisture content of the mat, wood dust usage, density and sanding are the mainly parameters influencing surface properties of the final products (Nemli *et al*. 2007).

In addition, wood fiber properties such as anatomical and chemical properties of fiber, fiber structure and strength, and composition of fiber are basic parameters affecting the properties of fiberboard when the wood is used as raw material (Ayrilmis 2008).

Generally, beech, oak (low quality) and pine are preferred to use as raw materials for the MDF manufacture in Turkey. These species can be used as a single or a mixture. Using the mixture of

species in the manufacture process is an important factor. The mechanical and physical properties of final products are affected by this factor (Akbulut *et al*. 2000).

Ash (*Fraxinus*), birch (*Betula*), lime (*Tilia*), spruce (*Picea*), larch (*Larix*), Douglas-fir (*Pseudotsuga*) fibers are known as high quality raw material for MDF manufacture (Akgül *et al*. 2007). Ash wood is used to manufacture bentwood, tool handles, baseball bats and tennis rackets due to its high elasticity, shock resistance and splitting resistance (Zhong *et al*. 2013).

OBJECTIVE

The objective of this study was to evaluate some properties such as surface roughness parameters, colour change, water absorption and thickness swelling of MDF panels manufactured using the different ratios of *Fraxinus excelsior* L. sawdust.

MATERIAL, METHOD, EQUIPMENT

In this study, pine fibers and ash sawdusts were used as raw materials. Urea formaldehyde was used as resin. Before panel manufacturing, the sawdusts were converted into small fiber size. The fibers and sawdusts were dried in a laboratory oven until they reach 2% moisture content. Sawdusts were mixed to fibers at the ratio of 10, 15, 20% by weight, and mats were manually formed using 13% urea formaldehyde. Later, mats were pressed at a temperature of 180 ^{o}C for 7 min in a press. Before the experiments, the panels were conditioned in a climatized room at 20 ^{O}C and 65% relative humidity. Panel type and contents of panel type were given in Table 1.

Table 1

Panel type and contents of panel type

Panel type	Contents
A	90% fiber +10% sawdust
B	85% fiber +15% sawdust
C	80% fiber + 20% sawdust
Control	100% fiber (without sawdust)

Surface Roughness

Surface roughness parameters of panel samples were determined by portable surface roughness instrument. The surface roughness parameters (R_a, R_z) were measured to evaluate surface roughness of the samples according to DIN 4768 (1990) standard.

Colour Change

Colour change measurement of panel samples was carried out by using a spectrophotometer, Minolta CM-2600d, according to the CIE *L*a*b** method (HunterLab 2008). Total colour change values (ΔE^*) were measured to determine the colour change of panel samples. In this measurement, control value of panel samples was taken as references.

Water Absorption and Thickness Swelling

Water absorption (WA) and thickness swelling (TS) of panel samples for 24h were determined according to EN 317 (1993) standard.

RESULTS AND DISCUSSION

Surface roughness

The surface roughness parameters (R_a, R_z) values of MDF panel groups were represented in Fig.1 and Fig. 2.

Fig. 1 shows that the R_a values of MDF panel samples changed depending on the ratio of sawdust. R_a values of panel samples increased with increasing the ratio of sawdust. The lowest R_a value was found to be 3,87 µm for control panel. The R_a values for panel types A, B, C were determined higher than the R_a value of control panel. The R_a value of panel type A manufactured with sawdust at the 10 % ratio had the lowest value (4,31 µm) among the all MDF panels manufactured with sawdust, whereas R_a value was the highest (6,67 µm) for panel type C. This could be reasoned from the differences in the structure, properties and size of raw materials. Akgül *et al*. (2012) found that the average R_a value of the manufactured panels increased with increasing the rhododendron fiber ratio in the mixture.

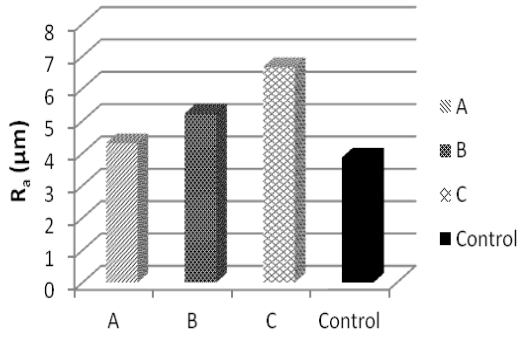

Fig. 1.
R_a values of samples.

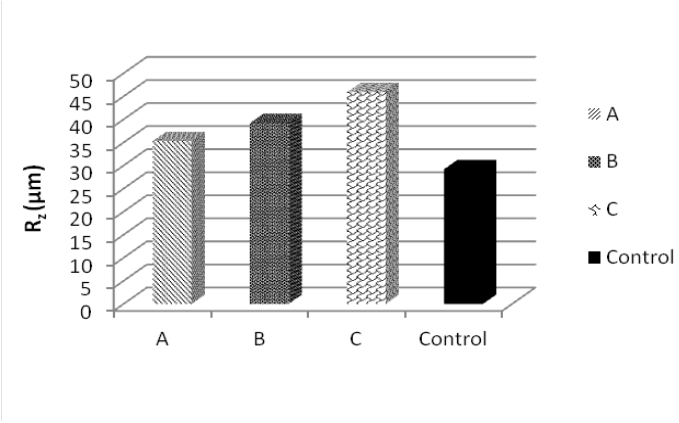

Fig. 2.
R_z values of samples.

Fig. 2 also shows that the similar trend was observed for R_z values of all MDF panel samples. The R_z values of panel samples increased with increasing the ratio of sawdust. The lowest R_z value was found to be 29,2 µm for control panel. All R_z values of panel types A, B, C manufactured with sawdust were found higher than that of control panel. The panel type C consisting of 20% sawdust had highest value (45,9 µm) among the other panel types consisting of 15%,10% sawdust, and control panel. The rougher surface was obtained from panel type C.

These results could be attributed to fiber and sawdust properties, sawdust size. Hiziroglu and Kosonkorn (2006) reported that the shape, height and width of the irregularities affect the surface quality of a final product.

Colour Change

The colour change *(ΔE*)* of panel groups is presented in Fig.3.

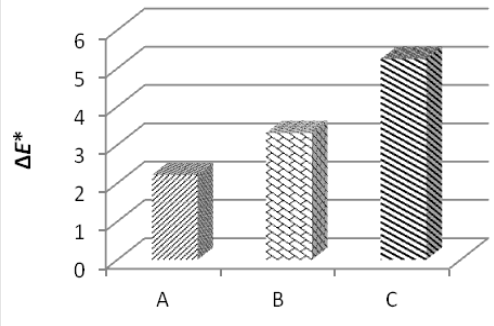

Fig. 3.
Colour change values of panel samples.

As can be seen from Fig.3 the Δ*E** values of MDF panel samples changed depending on the ratio of sawdust. The colour change (Δ*E**) values of panel samples increased with increasing the ratio of ash sawdust. The highest colour change value (5,24) was obtained with panel type C with 20% sawdust ratio. The lowest Δ*E** value (2,21) was found in the panel type A with 10% sawdust ratio.

Water Absorption and Thickness Swelling

The results of the water absorption and thickness swelling for 24 h of MDF panel samples depending on the ratio of sawdust were given in Fig.3. and Fig. 4.

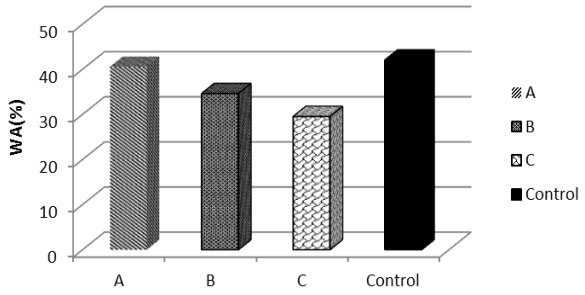

Fig. 3.
Water absorption values of panels.

As can be seen from Fig. 3, the WA values of MDF panel samples decreased with increasing the ratio of sawdust. The WA values of panel types A, B, C were found lower than the WA value of the control panel. The highest WA value was recorded from control panel. This value was found to be 42,05%. The lowest WA value was obtained to be 29,54% from panel type C manufactured with sawdust at the 20% ratio.

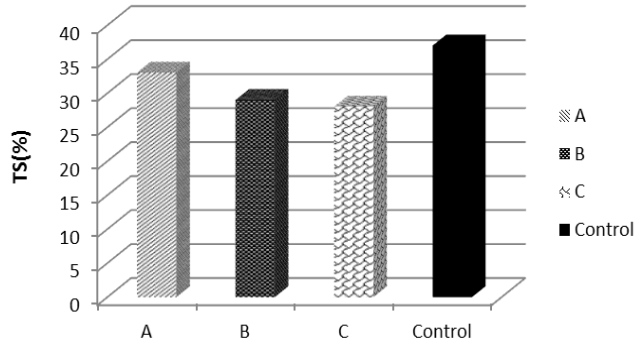

Fig. 4.
Thickness swelling values of panels.

Fig. 4 shows the TS values of MDF panel samples. The tendency of TS values was found similar to the WA values. It was determined that the TS values also reduced with increasing the ratio of sawdust. The highest TS value was found to be 37,04% for control panel. The panel type C had the lowest TS value. This value was recorded to be 28,43%.

Based on these findings (Fig.3 and Fig.4), it appears that the WA and TS values changed depending on the ratio of ash sawdust. The highest WA and TS values were determined from the control panels. Water absorption and thickness swelling values improved with increasing sawdust ratios. **This trend in WA and TS values could be attributed to raw material properties, sawdust size, the ratio of mixture of raw material species.** Akbulut *et al.*(2000) reported that the mixture of species in the panel manufacturing affects the physical and mechanical properties of products.

CONCLUSIONS

As a conclusion, it was found that the ratio of sawdust has effects on the panel properties. It is clear that all the values of MDF panels showed differences depending on the ratio of sawdust. While the surface roughness parameters (R_a, R_z), colour change (*ΔE**) values of panel samples increased with increasing the ratio of ash sawdust, the water absorption (WA) and thickness swelling (TS) values of panel samples decreased. The highest R_a, R_z, *ΔE** values were obtained from panel type C consisting of 20% ash sawdust. Especially, an improvement was observed in WA and TS values. The lowest WA and TS values were determined from the panel type C.

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EFFECT OF TIO_2 ON SOME PHYSICAL PROPERTIES OF WOOD POLYMER NANOCOMPOSITES (WPN)

Alperen KAYMAKCI
Kastamonu University, Faculty of Forestry, Forest Industry Engineering
Deparment of Wood Mechanics and Technology
Phone: +9 0366 280 1773 Fax: +9 0366 215 2316
E-mail: akaymakci@kastamonu.edu.tr

Emre BIRINCI
Kastamonu University, Arac Rafet Vergili Vocational High School, Depatment of Forestry.
Phone: +9 0366 362 2620 Fax: +9 0366 215 2316
E-mail: ebirinci@kastamonu.edu.tr

Turker GULEC
Artvin Coruh University, Faculty of Forestry, Forest Industry Engineering
Deparment of Wood Product Chemistry and Technology
E-mail: tgulec@artvin.edu.tr

Nadir AYRILMIS
Istanbul University, Faculty of Forestry, Forest Industry Engineering
Deparment of Wood Mechanics and Technology
E-mail: nadiray@istanbu.edu.tr

Abstract

In this study, some physical properties of polypropylene nanocomposites reinforced with nano TiO_2 were investigated To meet this objective, The nano TiO_2 was compounded with polypropylene with coupling agent in a twin screw co-rotating extruder and then the compounds were injection molded into the test specimens using an injection molding machine. The water absorption and thickness swelling properties of WPNs improved with increasing nano TiO_2 content. The use of maleic anhydride polypropylene had a positive effect on the pysical properties of the polypropylene nanocomposites reinforced with nano TiO_2. This work showed that the composites reinforced with nano TiO_2 could be efficiently used as decking products, due to satisfactory physical properties of the nanocomposites.

Key words: *nanocomposites; polypropylene; nano TiO_2; decking product; dimensional stability.*

INTRODUCTION

New kinds of bio-based building materials produce from renewable lignocellulosic resources, such as wood, are becoming more interesting and attractive. The principal reason for this can be related to global environmental challenges and the need for a sustainable development in society (Segerholm 2012).

Green development can only be probable when production uses renewable materials or materials recycled from wastes. As a consequence of the increasing demand for eco-friendly materials and the high cost of synthetic fibers, new bio-based materials containing natural fibers were developed (Kidalova et al. 2012, Kordkheili et al. 2013, Safdari et al. 2011).

To produce wood plastic composites (WPCs), wood residuals, e.g. sawdust, wood shavings or wood flour are mixed with a thermoplastic polymer and melt processed (or thermoformed) into its final shape, either as a continuous profile forming through extrusion or as a three dimensional form through injection molding. During recent decades, WPCs have rapidly increased their market share as a building material (Carus and Gahle 2008). Indeed, the addition of wood residuals as renewable natural filler in WPCs aims to produce a unique combination of high performance, great versatility, light weight, recyclability, biodegradability and processing advantages at favorable cost (Retegi *et al.* 2009; Kaci *et al.* 2009).

One of the main disadvantages linked to the use of renewable natural fibers is their moisture uptake when exposed to environmental conditions and its influence on the properties of composites. Data on the effects of moisture retention on physical properties of WPCs during long term service are crucial for their outdoor applications (Retegi *et al.* 2009). In order to improve the poor adhesion

between hydrophilic lignocellulosic material and hydrophobic thermoplastic matrices, chemical coupling agents and fiber modification techniques have been applied (Patel *et al*. 2012). Surface modification of lignocellulosic fibers with acetic anhydride or other chemical modification was employed by several researchers (Ozmen 2012). However, the most common application is the utilization of maleic anhydride grafted polypropylene (MAPP) and maleic anhydride grafted polyethylene (MAPE) for the polyolefins (Clemons 2002; Karakus *et al*. 2016).

On the other hand, nano science and nanotechnology have provided a new way to develop WPCs (Lu *et al*. 2006). Nanotechnology is a very promising area for enhancing the mechanical, physical as well as other properties of WPCs using nanosized fillers. In the WPCs, different types of filler are used for improving the mechanical, physical as well as other properties. The surface characteristics of nano materials play a vital role in their fundamental properties from phase transformation to reactivity (Song 1996).

OBJECTIVE

The goal of the present study was to investigate the effect of nano TiO_2 loading level (0,1, 2, 3, 4, and 5 wt%) on the some physical properties of wood polymer nanocomposites.

MATERIAL, METHOD, EQUIPMENT

Polypropylene (Borealis Incorp), nano TiO_2 (Grafen company), and coupling agent (Optim-425, MFI/190 °C; 2,16 kg = 120 g/10 min, density: 0,91 g/cm^3) were used in the experiments. Some physical and technic properties of nano TiO_2 are presented in Table 1.

Table 1

Specification of the TiO_2

Properties	**TiO_2**
Apperance	White Powder
Average Particle Size	10-25 nm
Purity	99.5 %
Surface Area	> 50 m^2/g

Pine wood flour (40 mesh) as lignocellulosic filler obtained from a commercial WPC manufacturer (sema wood) in Tekirdag, Turkey. Experimental design of the study is presented in Table 2.

Table 2

Experimental design of the study

WPN Groups	**Polypropylene (wt%)**	**Pine Wood Flour (wt%)**	**TiO_2 (wt%)**	**MAPP (wt%)**
A	50	50	0	3
B	50	50	1	3
C	50	50	2	3
D	50	50	3	3
E	50	50	4	3
F	50	50	5	3

Depending on the nanocomposite groups, granulated polymer, wood flour, nano TiO_2 and MAPP were mixed. Then this mixture was compounded in a laboratory scale twin-screw extruder at 40 rpm screw speed. Extruder temperatures were set as 170, 180, 185, 190, and 200°C for 5 heating zones. The extrudates were collected, cooled and granulated into pellets. Finally, pellets were injected at injection pressure between 5 and 6MPa with cooling time about 30s. The specimens were conditioned at a temperature of 23°C and relative humidity of 50%.

Measurement

Water absorption (WA) and thickness swelling (TS) tests were carried out according to ISO 62. Five replicates were used for each nanocomposites group. The weight and thickness of dried specimens was measured to a precision of 0.001mm. The specimens were then placed in distilled water and kept at room temperature. For each measurement, At the end of each immersion time, the

specimens were taken out from the water and all surface water was removed with a clean dry cloth. The specimens were weighed to the nearest 0.01g and measured to the nearest 0.001mm immediately. The measurements were terminated after the equilibrium thicknesses of the specimens were reached. The values of the WA and TS as percentages were calculated with Eq. (1) and Eq. (2).

$$WA = \frac{W(t) - .Wo}{Wo} x100 \quad (1)$$

where: WA is the water absorption (%) at time t, W_0 is the initial weight of the specimen, and W(t) is the weight of the specimen at a given immersion time t.

$$TS = \frac{T(t) - .To}{To} x100 \quad (2)$$

where TS is the thickness swelling (%) at time t, T_0 is the initial thickness of the specimen, and T(t) is the thickness of the specimen at a given immersion time t.

RESULT AND DISCUSSION

Figures 1 and 2 show the water absorption and thickness swelling percentages for the WPNs at different periods of immersion, which vary depending upon the nano TiO_2. In all the cases, the water absorption was found to increase with increasing time of immersion. Generally, the water absorption curves of WPNs increase with an increase in soaking time until equilibrium conditions are reached.

Table 3

Physcal properties of the nano TiO_2 reinforced wood polypropylene nanocomposites.

WPN groups[1]	Physical properties					
	Thickness swelling (%)			Water absorption (%)		
	1-day	7-days	28-days	1-day	7-days	28-days
A	0.81 (0.03)	1.75 (0.11)	1.83 (0.24)	0.47 (0.08)	0.75 (0.11)	0.89 (0.08)
B	0.77 (0.05)	1.69 (0.27)	1.74 (0.32)	0.46 (0.03)	0.75 (0.14)	0.85 (0.19)
C	0.74 (0.02)	167 (0.24)	1.72 (0.28)	0.44 (0.07)	0.72 (0.19)	0.81 (0.17)
D	0.69 (0.07)	1.64 (0.14)	1.70 (0.07)	0.40 (0.09)	0.68 (0.17)	0.78 (0.19)
E	0.65 (0.06)	1.60 (0.41)	1.65 (0.14)	0.37 (0.11)	0.67 (0.09)	0.74 (0.13)
F	0.63 (0.04)	1.55 (0.21)	1.61 (0.17)	0.34 (0.03)	0.60 (0.07)	0.69 (0.17)

[1] See Table 2 for WPC formulation.
The values in the parentheses are standard deviations.

Table 3 represented water absorption and thickness swelling ratio of wood polymer nanocomposites (WPN). As it is clearly seen, generally, water absorption and thickness swelling ratios increased with immersion time, reaching a certain value at saturation point, beyond which no more water was absorbed and the composites water content remained constant. The hydrophilic nature of wood flour causes the water absorption in manufactured WPNs. Because of constant wood flour content (50 wt%) in all designs, the different water absorptions and thickness swelling ratios among all manufactured nanocomposites can be attributed to the role of coupling agent and nano TiO_2. The water absorption and thickness swelling of the specimens decreased with increasing nano TiO_2 loading (Table 3). It seems that the barrier properties of nano TiO_2 inhibit the water permeation in the polymer matrix. This barrier property hinders water from going into the inner part of the nanocomposites (Kord 2014). Another reason for less water absorption could be the change in crystallinity of WPNs coupled by MAPP and existence of nano TiO_2 as a nucleating agent. It was reported that crystallinity of the WPNs is much greater than that of the corresponding WPNs without the MAPP modification (Ghasemi and Kord 2009). In addition to this, the nucleation efficiency and the

crystallinity of the hybrid composite can be improved at the presence of the nano reinforcing filler as a nucleating agent. As the crystalline regions are impermeable to penetration, the water absorption and thickness swelling are reduced in the nanocomposites (Ghasemi and Kord 2009; Kaymakci 2016).

The dimensional stability of the specimens was significantly improved by the incorporation of the MAPP. However, the positive effect of the MAPP on the dimensional stability of the specimens decreased at higher contents of the nano TiO2. The MAPP improves the interfacial adhesion between the pine wood flour, polypropylene and nano TiO_2, leading to less microvoids and fiber-polypropylene-carbon nanotube debondings in the interphase region. The chemical reaction of the hydrophilic hydroxyl groups of the lignocellulosics and acid anhydride groups of the MAPP, thus forming ester linkages reduce the number of free hydrophilic groups. This indicates that chemical bonding of hydroxyl groups of the pine wood flour with functional groups of the MAPP at the interface reduces water uptake of the specimens (Ayrilmis et al. 2012).

CONCLUSIONS

In this study, the effect of nano TiO_2 on some physical properties of wood polymer nanocomposites was investigated. By increasing the content of nano TiO_2, the dimensional stability of the nanocomposites was improved. The minimum water absorption and thickness swelling values were observed in nanocomposites made of 5 (%) of nano TiO_2. The findings obtained in this study clearly showed that water absorption and thickness swelling of the wood polymer nanocomposites increased with immersion time, reaching a certain value at saturation point, beyond which the water content of composites remained constant. The dimensional stability of the specimens was significantly improved by the incorporation of the MAPP. The MAPP improves the interfacial adhesion between the pine wood flour, polypropylene and nano TiO_2, leading to less micro voids and fiber-polypropylene-nano TiO_2 debondings in the interphase region.

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EFFECT OF WOOD CHIP SIZE ON HEMICELLULOSE EXTRACTION AND TECHNOLOGICAL PROPERTIES OF FLAKEBOARD

Nadir AYRILMIS
Department of Wood Mechanics and Technology, Forestry Faculty, Bahcekoy
Address: 34473, Sariyer, Istanbul, Turkey
Tel.: +82 33250 8324, Fax: +82 33256 8320, Email: nadiray@istanbul.edu.tr

Jin Heon KWON
Department of Forest Biomaterials Engineering, College of Forest and Environmental Sciences
Kangwon National University, 200-701, Chuncheon, Republic of Korea
*Dual Corresponding authors: Tel.: +90 212 338 2400, Fax: +90 212 338 2424
Email: kwon@kangwon.ac.kr

Tae Hyung HAN
Department of Forest Biomaterials Engineering, College of Forest and Environmental Sciences
Kangwon National University, 200-701, Chuncheon, Republic of Korea
Email: thhan212@kangwon.ac.kr

Abstract

Effect of chip size on the hemicellulose extraction of wood and technological properties of flakeboard was investigated. Three different sizes of wood chips were treated at 170°C for 90min in the hot water in a digester. After the treatment, the chips were washed and then dried. The uniform size flakes were produced from the control and treated chips using a flaker. The flakeboards were produced from the flakes using urea-formaldehyde resin. The results showed that the chip size had a significant effect on the chemical properties of hydrothermally treated wood under pressure in hot water. As the chip size was decreased, the amounts of the extractives and hemicelluloses decreased in the wood while the amounts of the cellulose and lignin increased. As for the physical properties of the flakeboards, thickness swelling and water absorption significantly decreased with decreasing chip size. There were significant differences in the flexural strength and internal bond strength of the flakebords produced from untreated and treated chips. The internal bond strength of the flakeboards improved with decreasing chip size. It was concluded that the flakeboards produced from the flakes of hydrothermally treated chips (thickness: 7.4mm, width: 9.2mm, and length: 38.5mm) gave the optimum physical and mechanical properties.

Key words: *chemical properties; flakeboard; hydrothermal treatment; mechanical properties; physical properties; wood.*

INTRODUCTION

Wood is a complex polymeric material constituted mainly of cellulose, hemicelluloses and lignin, with a minor proportion of extractives. Hemicellulose represents 20-35% of wood structural polymer and is the most unstable and hydrophilic polymer of wood (Godvarti 2005). Hydroxyl groups in hemicellulose are readily form hydrogen bonds with water. This results in a high rate of water uptake and thickness swelling in the wood and wood-based panels. Using the wood particles with a less hydrophilic property can improve the dimensional stability of wood-based panels. However, a major drawback of thermal-treatment is that wood strength is reduced (Ayrilmis et al. 2009).

Hydrothermal treatment (heating of the wood under wet conditions) is one of the most effective ways to improve dimensional stability of the wood. In this process the hemicellulose and water soluble extractives are removed from wood (Charani et al. 2007; Hill 2006; Burgos and Rolleri 2012). No chemical other than hot-water is used in this process, thus hydrothermal treatment is considered as environmentally friendly technology (Li et al. 2013). After the extraction, wood surfaces are covered with lignin components, which are less hydrophilic (Hosseinaei et al. 2011). In addition, decreasing the hydroxyl groups due to the removal of hemicelluloses reduces the hygroscopic sites of wood. This results in enhancement in the dimensional stability in wood and wood-based panels.

In the literature, effect of chip size on the extraction of hemicelluloses in wood was investigated by Li et al. (2013). They reported that the total extraction yield, as well as the yields of hemicelluloses and monosaccharides, increased clearly with decreasing particle size. However, based on the

extensive literature search, there was no any study on the relationship between hydrothermally treated chip size and technological properties of wood-based panel.

OBJECTIVE

The objective of the present work was to investigate the influence of wood chip size on the hemicellulose extraction and technological properties of the flakeboard. The flakes prepared from hydrothermally treated wood chips and flakeboards were produced at laboratory. From the test results, the flakeboard manufacturers can use the proper chip size for the hydrothermal treatment before the production of the flakeboard.

MATERIAL, METHOD, EQUIPMENT

Wood material and resin

Korean pine (*Pinus koraiensis S. et Z.*) log was cut into the roundwood parts. Then these parts were divided to the wood discs using saw mill. The discs without bark were cut into *longitudinal* strips using a table saw. The chips to be treated in digester were prepared from these strips. The thickness (38.5mm) of the wood discs was equal to the length of the chips. The width and thickness of the chips were determined by adjusting the *distance* from the saw blade to the miter gauge so that three different chip sizes were obtained. The length of the chips were constant while the length and width of the chips were gradually increased. The dimensions of chips size are presented in Fig 1.

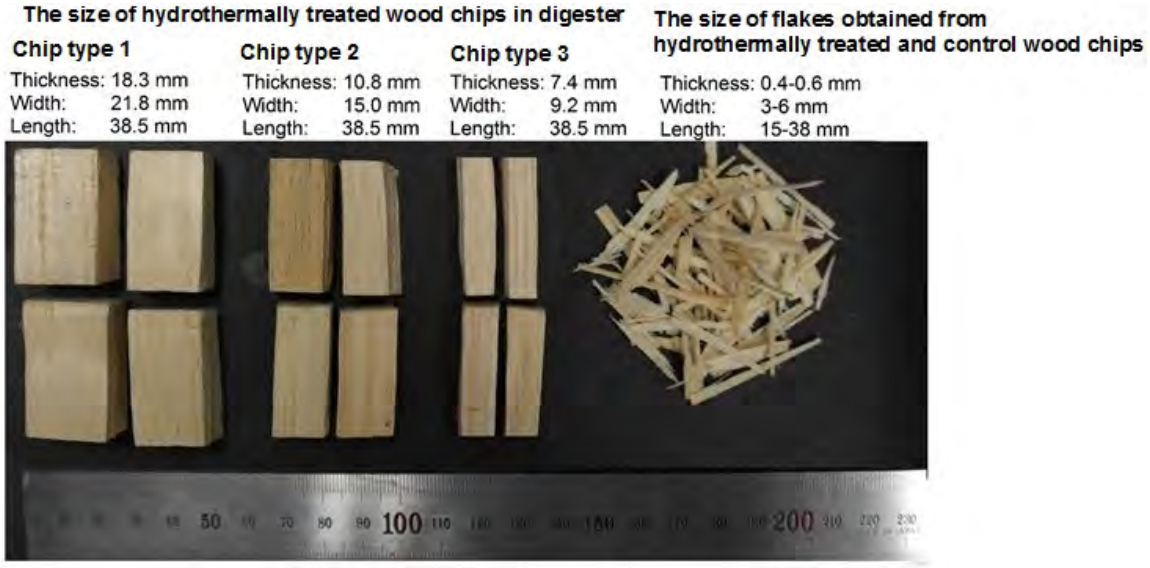

Fig. 1.
The sizes of wood chips used for the hydrothermal treatment.

A commercial liquid urea-formaldehyde (UF) (E1 class) resin with 63.3wt% solid content was supplied by a commercical wood-based panel manufacturer located in Republic of Korea. The viscosity and pH value of the resin were 0.185Pa.s and 8.0, respectively. No wax or other hydrophobic substance was used in the flakeboard manufacture. As a hardener, 1wt% of *ammonium chloride* (NH_4Cl) solution with 20% solid content based on the resin solid content was added into the resin.

Hydrothermal treatment of wood chips

The twin digester (GIST co. Ltd.) was used for hydrothermal treatment of different size chips. The water of 10L and wood chips of 2kg were put into the digester and then hydrothermally treated at 170°C for 90min. Three replications for each type of treatment, totally 6kg chips, were performed. The pressure gauge of the digester was between 0.18MPa and 0.20MPa. The hydrothermal treatment process of the chips is presented in Fig. 2.

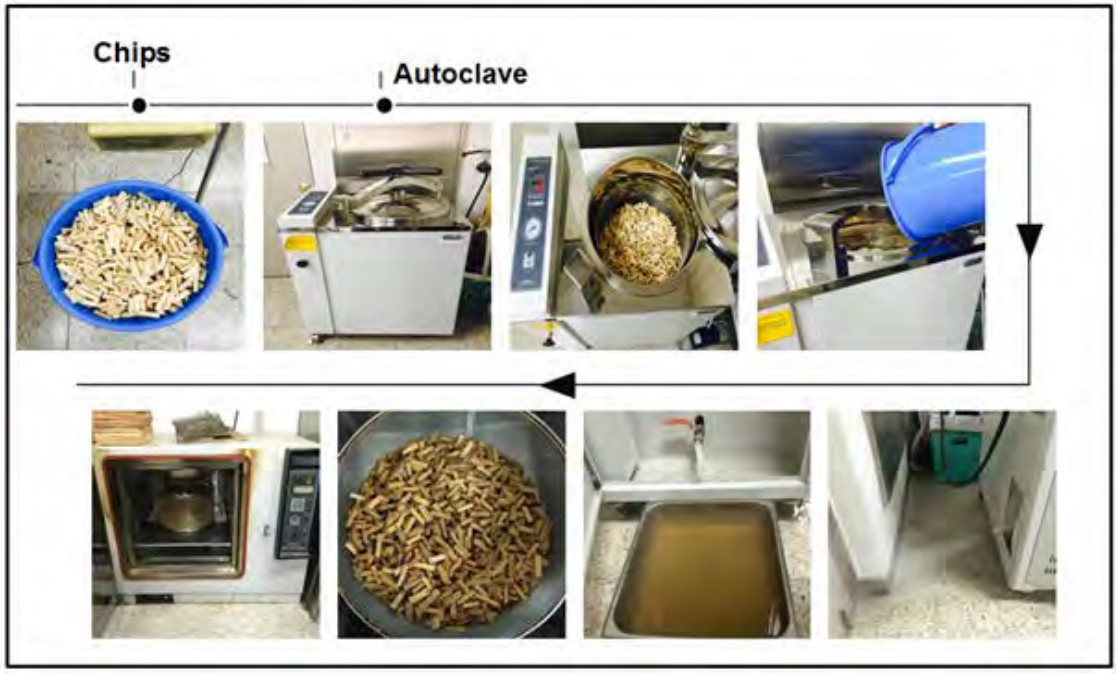

Fig. 2.
Hydrothermal treatment of chips.

The treated chips were then washed with water, air dried, and finally oven-dried at 70°C for 48h. This procedure was individually applied for each chip sizes. The chips *were cut* by a laboratory *disk* flaker (*Pallmann*) to produce the flakes. The thickness, width, and length of the flakes were 0.4-0.6mm, 3-6mm, and 15-38mm, respectively. Before the flakeboard production, the flakes were dried in an oven at 90°C for 24h to moisture content of average 2-3% based on the oven-dry solid weight of the flake. The wood chip size and hydrothermal treatment of wood chips are given in Table 1.

Table 1

The hydrothermal treatment of chips

Wood chip code	Chip size			Hydrothermal-treatment	
	Thickness (mm)	Width (mm)	Length (mm)	Temperature (°C)	Duration (min)
Control (A)	7.4	9.2	38.5	-	-
B	18.3	21.8	38.5	170	90
C	10.8	15.0	38.5	170	90
D	7.4	9.2	38.5	170	90

Determination of the chemical properties of the wood chips

For the determination of the chemical properties of hydrothermally treated wood chips having different sizes, 10g wood flour (40 mesh) was prepared using a grinder. Holocellulose analysis was done according to Wises's sodium chlorite method, cellulose was determined by Kurschner-Hoffner's nitric acid method, and lignin content was determined as acid-insoluble Klason lignin (1962). Hemicellulose content was found by subtracting cellulose content from holocellulose content. Three samples were used for determination of the chemical properties. Total extractives were determined according to TAPPI T 204 om-88 (1988) standard procedure in soxhlet apparatus. Two solvents were successively used each time for 6 hours as follows: the mixture of ethanol with benzene (1:2 v/v). The weight loss of the flakes was calculated after the hydrothermally treatment under pressure in hot water.

Production of flakeboards

The flakes were glued with the liquid UF resin with the hardener to have a 10wt% resin based on the oven-dry weight of flake in the resin blender. The flakes were hand-formed into randomly oriented mats with dimensions of 290mm×290mm×10mm. The flakeboard mats were hot-pressed at 2.8MPa pressure, 170°C, and 7min. A total of 12 flakeboards, 3 flakeboards for 4 series of flakeboards (from type A to type D), were produced at wood composite pilot laboratory (Fig. 1). Prior to testing, the

specimens were conditioned to constant mass at a temperature of 20°C and a relative humidity of 65%.

Determination of physical and mechanical properties

Physical and mechanical properties of the flakeboards were determined according to Korean Standard (KS) F 3104 (2002). One day thickness swelling (TS) and one day water absorption (WA) tests were performed on the ten specimens with dimensions of 50mm×50mm×10mm. The density test was performed on the ten specimens with dimensions of 100mmx100mmx10mm. A total of ten specimens (200mm×50mm×10mm) (5 // and 5 ⊥ to the flakeboard surface) were tested for each type of flakeboard to determine the bending strength (MOR) and modulus of elasticity (MOE). Internal bond (IB) test was performed on the ten specimens with dimensions of 50mm×50mm×10mm.

Statistical analysis

An analysis of variance, ANOVA, was conducted ($p<0.05$) to evaluate the effect of hydrothermally treated chip size on the chemical properties of wood and technological properties of the flakeboard. Significant differences among the average values of the flakeboard groups were determined using Duncan's multiple range test.

RESULTS AND DISCUSSION

Weight Loss and Chemical Composition of Wood Chips

The influence of the hydrothermal treatment on the weight loss and chemical properties of different sizes of the wood chips was given in Table 2. As the hydrothermal treatment parameters were kept constant, the weight loss of the chips considerably increased as the chip size decreased. The total weight losses of the chips B, C, and D were found to be 15.45%, 17.24% and 20%, respectively. The highest decrease was found in the hemicellulose content and followed by the extractives. For example, as compared to untreated control chips, the hemicellulose and extractives of the chip type D decreased from 21.2% to 4.1% and 9.4% to 4.9%, respectively. On the other hand, the amounts of lignin and cellulose considerably increased with decreasing size of the treated chips. The amount of the lignin of the control chips increased from 38.0 to 51.9% when they was treated at 170°C for 90min in the digester. Similarly, the cellulose content of the chips increased from 31.2 to 39.0%. The increase in the weight loss of hydrothermally treated biomass has been reported previously elsewhere (Yoon and Heiningen 2010; Kwon and Ayrilmis 2016; Dos Santos et al. 2014).

Table 2

Weight loss and chemical composition of the control and treated chips

Wood chip code	Total weight loss (%)	Chemical composition of wood chips (%)			
		Lignin	Cellulose	Hemicellulose	Extractives
Control (A)	0	38.0	31.2	21.2	9.4
B	15.45	42.0	36.9	12.8	8.4
C	17.24	46.9	37.7	8.7	6.6
D	20.0	51.9	39	4.1	4.9

Hydrolysis of hemicellulose is the main reason for the weight loss in the flakes. Hot water under pressure penetrates into the cell structure of wood, hydrates, and removes hemicellulose (Wyman et al. 2005). Thermal exposure in the digester can alter the hemicelluloses structure because arabinan and galactan, each a side-chain component of the hemicelluloses, which tends to be more degraded as the chip size is decreased. The results showed that a decrease in the chip size resulted in better penetration of the hot water into the cell walls, higher hydrolysis of hemicellulose, and higher weight loss. Some parts of extractives (hot water soluble extractives) removed during the treatment as a part of weight loss.

Hot water under pressure penetrates into the cell walls, cleaving acetyl groups of hemicellulose and generating acetic acid, which catalyzes hydrolysis of the hemicellulose and the formation of monomeric sugars (Yildiz and Gumuskaya 2007). The concentration of organic acids, in particular for acetic acid, generated from cleavage of acetyl groups of hemicellulose increased with decreasing chip size. Larger chips (Type 1 in Fig. 1) caused a decrement in the penetration of hot water into the lumens and cell wall, in particular into the center of the chip, as compared to the smaller chip (Type 3). This could be a reason for lower weight loss in larger chips through decreased accessibility of hot water to polymers in the cell wall.

Physical and Mechanical Properties of Flakeboards

The results of physical properties of the flakeboards are given in Table 3. A slight increment in the density of the flakeboards was determined. The flakeboards produced with untreated flakes showed the highest amounts of both WA and TS which were significantly different from those of the flakeboards produced with any of the extracted samples. The TS and WA of the flakeboards significantly decreased as the size of hydrothermally treated chips decreased. For example, the average TS and WA of the flakeboards decreased from 54.5 to 14.8% and 77.1 to 44.1%, respectively, depending on hydrothermally treated chip size (Table 3). The control group had the highest TS and WA values while the flakeboard type D which had the lowest size had the lowest TS and WA values. The significant differences ($p<0.05$) among the flakeboard types from A to D are shown by letters in Table 3. A sharp decrease was observed in the TS and WA values of the flakeboards produced with the flakes obtained from chip type B as compared to the control flakeboard.

Table 3

Physical and mechanical properties of flakeboards

Flakeboard code	Physical properties			Mechanical properties		
	Density (g/cm^3)	TS (%)	WA (%)	MOR (N/mm^2)	MOE (N/mm^2)	IB (N/mm^2)
A	0.79 a (0.02)	54.5 a (2.7)	77.1 a (2.9)	15.3 a (1.4)	2415.4 a (224)	0.15 a (0.04)
B	0.82 a (0.03)	20.9 b (1.8)	58.1 b (2.1)	15.1 a (1.5)	2371.5 b (260)	0.19 a (0.05)
C	0.81 a (0.04)	15.8 c (1.2)	52.6 c (1.7)	14.6 a (1.0)	2334.1 b (274)	0.20 a (0.06)
D	0.82 a (0.03)	14.8 c (1.5)	44.1 d (1.4)	14.5 a (1.2)	2329.7 b (255)	0.18 a (0.05)

MC: moisture content. TS: thickness swelling. WA: water absorption.
MOR: modulus of rupture. MOE: modulus of elasticity. IB: internal bond strength.
The values in the parentheses are standard deviations.
Groups with same letters in column indicate that there is no statistical difference ($p<0.05$) between the specimens according to Duncan's multiply range test.

The significant differences in the water resistance of the flakeboards were mainly due to the degradation rate of the hemicelluloses depending on the chip size. Hemicelluloses are very hydrophilic compounds of wood cell and the most heat sensitive polymers of wood components hydrolyzed during the hydrothermal treatment. It is known that during the hydrothermal treatment of wood, carbonic acids, mainly acetic acid, are formed as a result of cleavage of the acetyl groups of hemicelluloses. These changes in the hemicelluloses can decrease the hygroscopicity of the flakes, which consequently results in lower TS and WA values for the flakeboards. Lignin deposits on the surface of the treated flakes after the extraction (Hosseinaei et al. 2015), which has *hydrophobic character,* may also contribute to decreased hygroscopicity of wood flakes.

The bending properties of the flakeboards are presented in Table 3. The MOR and MOE slightly decreased as the chips were hydrothermally treated. The statistical analysis results showed that there were no significant difference in the MOR and IB strength values between the control flakeboard and treated flakeboards. However, this was not observed for the MOE values. The MOE values of the flakeboards produced with three different sizes of treated chips were significantly ($p<0.05$) lower than that of the control flakeboard. The control group had the highest MOR ($15.3 N/mm^2$) and MOE ($2415.4 N/mm^2$) while the flakeboard type D had the lowest MOR ($14.5 N/mm^2$) and MOE ($2329.7 N/mm^2$).

The main reason for the lower bending properties of the flakeboard is the degradation of hemicelluloses, which are less stable to heat than cellulose and lignin. Soluble acidic chemicals such as formic acid and acetic acid formed by the degradation of the hemicellulose are mainly responsible for the mechanical properties of wood. In addition the soluble acids can break down the long chain cellulose (Sundqvist 2004), which reduce the strength of the wood. Similar results were observed in some previous studies on wood (Burgos and Rolleri 2012; Charani 2007; Dos Santos et al. 2014) and wood-based panels (Kwon and Ayrilmis 2016; Hosseinaei et al. 2011; Ayrilmis et al. 2011).

The IB strength of the flakeboards is given in Table 3. Apart from bending properties, the IB strength of all the flakeboards produced with the flakes of hydrothermally treated chips were found to be slightly higher than that of the control flakeboard. There was no significant difference in the IB values between the control flakeboard and treated flakeboards. The IB strength of the control

flakeboard was found to be 0.15N/mm^2. As compared to the control flakeboard, it was observed in an increase in the IB strength for the flakeboard types B (0.19N/mm^2) and C (0.20N/mm^2) types. However, a slight decrease was observed in the IB strength (0.18N/mm^2) of the flakeboard type D, but it was higher than that of the control flakeboard.

The enhancement in the IB strength of the flakeboards produced with flakes of hydrothermally treated chips could be related to the decrement in the weight loss of the flakes and thereby increment in the compression ratio in the flakeboard mat during the hot-pressing. A higher compression ratio can increase the magnitude of the bonding area between the flakes and subsequently improved IB strength (Ajayi 2000). In addition, the increased compaction ratio decreases the internal void volume which can hinder the migration of moisture into the flakeboard and improve the water resistance.

The hydrothermal treatment decreased the extracive content of the flakes as shown in Table 2. Extractives have a significant effect on resin bonding performance of wood (Nemli and Colakoglu 2005; Nemli and Aydin 2007; Ayrilmis et al. 2009). In previous studies, it was reported that extractives negatively affect the resin bond between wood particles. For example, Ayrilmis et al. (2009) reported that ethanol- and water-soluble extractives had a significant effect on the UF resin gel time which played major roles in determining resin bond-quality. The extractives and moisture from the flakes can make gas pressure in the flakeboard during the hot pressing and then the gas pressure can negatively affect the resin bond between the flakes. The lower h*ygroscopic* capacity and lower extractive content of hydrothermally treated flakes can reduce the gas pressure in the flakeboard, which do not have negative effects on the resin bond strength between the flakes, as compared to the control flakeboard having higher hygroscopic capacity and higher extractive contents.

CONCLUSIONS

The results of the present study showed that the chip size had a significant effect on the chemical properties of hydrothermally treated wood. As the chip size was decreased, the amounts of the extractives and hemicelluloses decreased while the amounts of the cellulose and lignin increased. As for the physical properties of the flakeboards, thickness swelling and water absorption of the boards significantly decreased with decreasing chip size. As the destruction rate of hemicelluloses increased the hygroscopic sites of wood decreased, which resulted in a higher water resistance. However, the flexural properties of the flakeboard decreased with decreasing with chip size, but it was not significant. The internal bond strength of the flakeboard improved with decreasing chip size. This showed that the resin bond between the flakes enhanced as the wood was hydrothermally treated in a digester. Based on the findings of this study, it was concluded that the chip type D had the optimum size (thickness: 7.4mm, width: 9.2mm, and length: 38.5mm) in terms of physical and mechanical properties of the flakeboards. The results showed that hydrothermal treatment of wood is an effective method to enhance serviceability of flakeboard.

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INFLUENCE OF THE CONTENT OF LIGNOSULFONATE ON PHYSICAL PROPERTIES OF MEDIUM DENSITY FIBERBOARD

Viktor SAVOV
University of Forestry – Sofia, Faculty of Forest Industry
10 Kliment Ohridski blvd. 1797 Sofia, Bulgaria
Tel: 00359 885 091198, E-mail: victor_savov@ltu.bg

Julia MIHAILOVA
University of Forestry – Sofia, Faculty of Forest Industry
10 Kliment Ohridski blvd. 1797 Sofia, Bulgaria
Tel: 00359 888 770216, E-mail: jmihajlova@ltu.bg

Abstract

One of the essential shortcomings in the production of MDF is the existence of formaldehyde emissions from boards. This could be overcome by replacement of the currently used synthetic binders. Lignin is a natural binder in wood. Globally, at laboratory level, there are numerous studies on the use of enzyme lignin as a binder for MDF. In these studies were observed some significant shortcomings that could be overcome with the use of lignosulfonates.

In this article is presented a study on the influence of the content of lignosulfonate in the composition of MDF made from hardwood tree species on their physical properties. The boards were produced with only 5% content of urea-formaldehyde resin and alteration in the content of calcium lignosulfonate from 0 to 20%.

The approximating functions for the influence of the content of lignosulfonate on physical properties of MDF were derived. On that base is made analysis with proper conclusions and recommendation for optimal content of calcium lignosulfonate in the composition of fiberboard.

Key words: *MDF; urea-formaldehyde resin; formaldehyde emission; calcium lignosulfonate.*

INTRODUCTION

As a material fiberboard is characterized by significantly better physical and mechanical properties in comparison with particleboards. Therefore, worldwide production of fiberboard is greater than the production of particleboard (FAO). It should be said that the increase in the production volume of fiberboard is mainly due to the increased production of Medium Density Fiberboard (MDF), which occupy a total of 78% of the overall production. This trend is also characteristic for Europe, production of wet processed fiberboard is replaced with a dry processed one. The advantages of the production by a dry method, in comparison with a wet, principally is in the ability to produce boards with a thickness greater than 8 mm and with two facial surfaces. However, there is one very important advantage of wet processed fiberboard, which is that they are eco-friendly. It means that in those boards there is practically no free formaldehyde. This determines the key feature for improving the technology for the production of MDF, a particular reduction or even replacing of traditionally used synthetic binders (urea-formaldehyde, melamine-formaldehyde, phenol-formaldehyde resins, etc.) and thus reducing the emissions of free formaldehyde from boards.

Interests in this field are attempts, though still in the laboratory stage, to be produced MDF by adding enzyme lignin, which acts as a binder for the boards (Zouh et al. 2011). In this way were obtained eco-friendly, nontoxic, boards. A team of Spain (Mancera et al. 2011) conducted similar studies. The content of enzymatic lignin was increased to 20%. In this case have been reported very good results, as the bending strength of the boards has reached more than 50 $N.mm^{-2}$, and swelling in thickness is less than 2%. Nevertheless, it should be stressed that the produced boards are with density more then 1300$kg.m^{-3}$.

A team from Malaysia (Nasir et al. 2014) with the addition of up to 30% enzyme lignin in fiberboard composition obtained in laboratory conditions MDF, conforms to the requirements of active standards (EN 622-5).

In all cited above studies there are several major drawbacks. The enzyme lignin is difficult for production and its price is high. Therefore, application of enzyme lignin as a binder will significantly increase the cost of MDF production. The lignin is not water-soluble and is used in the boards as an amorphous solid, which leads to major difficulties from a technological point of view. For the activation

of the enzyme lignin is required increased temperature of hot-pressing and extended duration of the process.

Partially these shortcomings can be overcome by the use of lignosulphonate as a binder for MDF. Lignosulfonate take precedence over hydrolysis or enzymatic hydrolysis lignin that it is water-soluble and can be imported in the form of solutions in pulp mass. They are a waste product from the production of sulphate cellulose. At present, lignosulfonates are used in the woodworking industry primarily as a binder conferring additional mechanical stability of the pellets.

MATERIALS AND METHODS

The main goal of this study is to be determined the influence of the content of calcium lignosulfonate in the composition of MDF on their physical properties. To be fulfilled this goal in laboratory conditions they were produced MDF containing urea-formaldehyde resin of 5% and with variation in the content of calcium lignosulfonate from 0 to 20% with increment of 5%. The boards were produced with a density 850kg.m-3. For the production of MDF in laboratory conditions was used thermo-mechanical pulp of common tree species in Bulgaria – total content of beech (*Fagus silvatica* L) and cerris oak (*Quercuss cerris* L) of 80% and 20% content of poplar (*Populus alba* L). Wood fiber mass was with moisture content of 11%.

It was used calcium lignosulfonate with characteristic as follow: calcium – up to 6%; reduced sugars – up to 7%; dry content – 93%; acid factor in a 10% solution - pH = 4,3±0,8; bulk density – 550kg.m^{-3}.

In order to more uniform distribution and easier activation of the calcium lignosulfonate it is inserted into the pulp in the form of a solution having a concentration of 30%, Fig.1.

Fig. 1.
Calcium lignosulfonate

The physical properties of MDF were determined by standard methods (EN 316; EN 317; EN 323). For each property were used in eight test specimens per board. And main statistical parameters (average, standard deviation, probability) were calculated.

The data were processed by the methods of regression analyze and it was displayed approximating function to the influence of the content of calcium lignosulfonate on the MDF properties.

On the basis of experimental data obtained by means of measurements, the values of the approximating function for different values of the argument were determined. This problem is successfully solved by using the least squares method, with regression equation of the type:

$$\hat{Y} = \sum_{i=0}^{k} b_i f(\tilde{x}) = b^T f(\tilde{x}) \quad (1)$$

where: $b^T = (b_0, b_1, \ldots, b_k)$ is a (k + 1)-dimensional vector of the unknown coefficients in the equation;

$\hat{y}$ – the predicted value of the output quantity;

$f^T(\tilde{x}) = [f_0(\tilde{x}), \ldots, f_x(\tilde{x})]$ is a (k + 1)-dimensional function of the vector of input variables $\tilde{x}$ being derived.

In the case of the least squares method, the polynomial of best root-mean-square approximation of given degree coincides with the interpolation polynomial.

As a criterion for approximation accuracy, the coefficient of determination is used (Trichkov 2015):

$$R^2 = 1 - \frac{\sum_{i=1}^{N}(y_i - \hat{y})^2}{\sum_{i=1}^{N}(y_i - \bar{y})^2} \quad (2)$$

RESULTS AND DISCUSSION

The summarized results for the properties of MDF, with different participation of lignosulfonate are presented in Table. 1.

Table 1

Experimental results for physical and mechanical properties of MDF

Board №	Content of calcium lignosulfonate *Px*, %	Density ρ, kg.m^{-3}			Swelling in thicknesses *Gt*,%			Water absorption *A*,%		
		Average	STDV	*P*-value	Average	STDV	*P*-value	Average	STDV	*P*-value
1	0	843.96	89.05	0.037	53.76	7.14	0.047	88.01	10.94	0.044
2	5	845.98	44.97	0.019	45.23	5.86	0.046	76.63	3.86	0.018
3	10	836.18	31.17	0.013	41.88	5.18	0.044	72.47	6.93	0.039
4	15	839.19	68.46	0.029	23.04	2.94	0.045	63.14	4.82	0.027
5	20	844.45	66.28	0.028	22.25	3.06	0.049	61.16	6.44	0.037

Analysis of experimental results for water absorption of MDF

The dependence of water absorption of MDF of the content of lignosulfonate over a range of variation of 0 to 20%, is described by the equation of the regression, approximating function, of the type:

$$\hat{A} = 87.67 - 2.12.P_x + 0.04.P_x^2 \text{ [\%]} \quad (3)$$

where: $\hat{A}$ is the predicted value for water absorption, %;

Px – content of calcium lignosulfonate in MDF, %.

The equation is characterized by the coefficient of determination R^2 = 0.98.

Figure 2 illustrates the variation of water absorption of MDF in function dependence from the content of lignosulfonate.

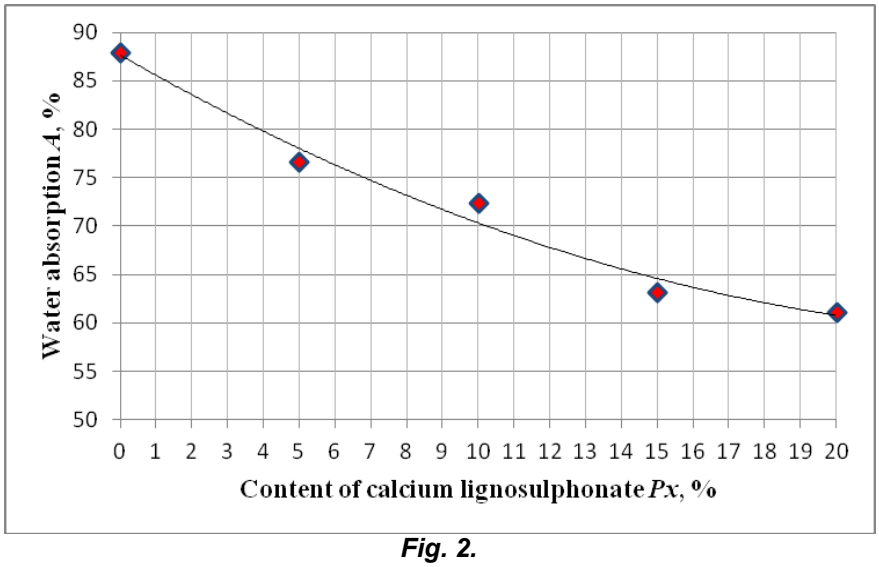

Fig. 2.

Variation of water absorption of MDF in dependence from the content of calcium lignosulfonate.

In the property water absorption is observed improvement with increasing the content of lignosulfonate from 0% to 20%, respectively, the value of the property is 88% at 0% content of lignosulfonate and 61% at 20% content of lignosulfonate in MDF.

There are two major declines in the values of the water absorption with the addition of 5% lignosulfonate, and with an increase the content of lignosulfonate from 10% to 15%. Values of the water absorption at 15% and 20% content of lignosulfonate are commensurate.

Analysis of the experimental results for swelling in thickness of MDF

The dependence of the swelling of MDF in thickness of the content of lignosulfonate over a range of variation of 0 to 20%, is described by the equation of the regression, approximating function, of the type:

$$\hat{G}_t = 54.27 - 1.70.P_x \text{ [\%]}, \quad (4)$$

where: $\hat{G}t$ is predicted value for swelling in thicknesses, %;

Px – content of calcium lignosulfonate in MDF, %.

The equation is characterized by the coefficient of determination $R^2 = 0.92$.

Figure 3 illustrates the variation of swelling in thicknesses of MDF in function dependence from the content of lignosulfonate.

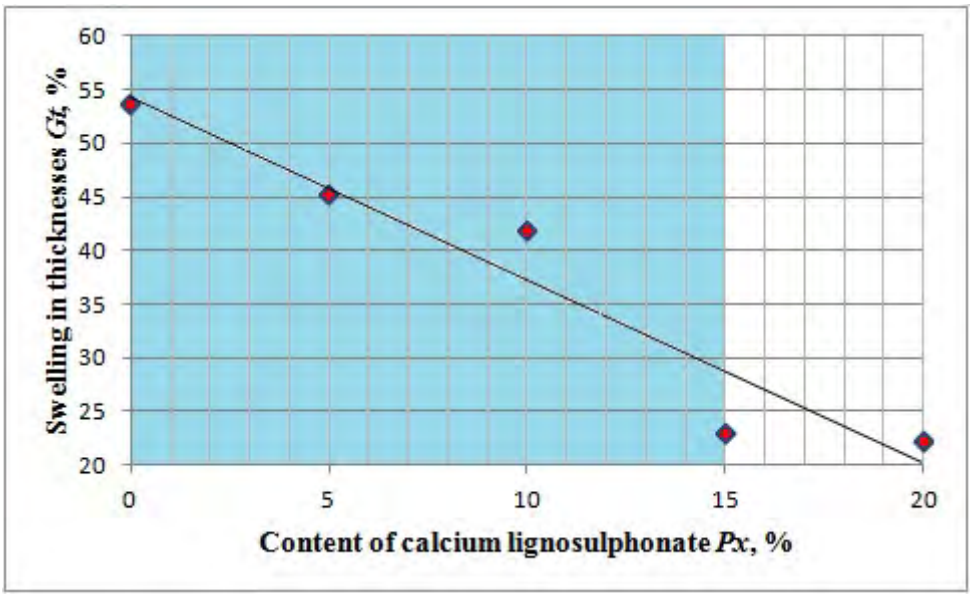

Fig. 3.
Variation of swelling in thicknesses of MDF in dependence from the content of calcium lignosulfonate.

With increasing the content of lignosulfonate from 0% to 20% swelling in thicknesses is improved, i.e. is reduced. It is not observed inflection point. In general, with the addition of lignosulfonate, swelling in thickness of MDF is reduced from 54% to 22% or the improvement of the property is exactly twice. This indicates that there is a lot of potential for the use of lignosulfonate in MDF intended for environment with high humidity.

The most significant improvement in the property was observed with an increase in the content of lignosulfonate from 10% to 15%. With increasing the content of lignosulfonate from 15% to 20%, there was no significant improvement in swelling in thickness of MDF.

Under the conditions of the experiment MDF does not meet the requirements for for use in humid conditions (EN 622-5).

On Figure 2 with light blue is marked the area where MDF does not meet the requirements for swelling in thicknesses for use in a dry conditions. The requirements for the swelling in thicknesses are implemented under a lignosulfonate content of 15 % or more. Therefore, the recommended content of lignosulfonate, wherein content of the urea-formaldehyde resin from 5% in MDF, is from 15 to 16%. In that content of calcium lignosulfonate are achieved requirements for MDF for load-bearing boards and use in dry conditions.

CONCLUSIONS

As a result of the conducted study on the influence of the participation of lignosulfonate in composition of MDF on its physical properties, can be made the following conclusions:

1) The addition of lignosulfonate in the composition of MDF leads to improvement, respectively, lowering the water absorption and swelling in thickness of the boards.
2) The most significant improvement of these properties is observed with increasing of the content of lignosulfonate by ten to fifteen percent;
3) It is not justified from a technological and economic standpoint to increase the content of lignosulfonate up to 20%.
4) MDF containing calcium lignosulfonate from fifteen to sixteen percent meet the requirements for load-bearing boards and are used in dry conditions and those boards are with only 5% content of urea-formaldehyde resin.

In conclusion, the use of calcium lignosulfonate is the perspective method for replacing the currently applied synthetic binders in MDF. This would make it possible to be reduced harmful emissions of the boards. Research in this area should be intensified by study of the influence of factors such as temperature and duration of hot-pressing, concentration of the lignosulfonate solution etc.

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INFLUENCE OF THE CONTENT OF LIGNOSULFONATE ON MECHANICAL PROPERTIES OF MEDIUM DENSITY FIBERBOARD

Viktor SAVOV
University of Forestry – Sofia, Faculty of Forest Industry
10 Kliment Ohridski blvd. 1797 Sofia, Bulgaria
Tel: 00359 885 091198, E-mail: victor_savov@ltu.bg

Julia MIHAILOVA
University of Forestry – Sofia, Faculty of Forest Industry
10 Kliment Ohridski blvd. 1797 Sofia, Bulgaria
Tel: 00359 888 770216; E-mail: jmihajlova@ltu.bg

Abstract

One of the essential shortcomings in the production of MDF is the existence of formaldehyde emissions from boards. This could be overcome by replacement of the currently used synthetic binders. Lignin is a natural binder in wood. MDF find mainly used in the production of furniture and furnishings. For that their application essential in view of the suitability of the boards are their mechanical properties.

In this article is presented a study on the influence of the content of lignosulfonate in the composition of MDF made from hardwood tree species on their mechanical properties. The boards were produced with only 5% content of urea-formaldehyde resin and alteration in the content of calcium lignosulfonate from 0 to 20%.

The approximating functions for the influence of the content of lignosulfonate on mechanical properties of MDF were derived. On that base is made analysis with proper conclusions and recommendation for optimal content of calcium lignosulfonate in the composition of fiberboard.

Key words: *MDF; urea-formaldehyde resin; formaldehyde emission; calcium lignosulfonate.*

INTRODUCTION

Medium Density Fiberboard (MDF) is wood based engineered material which is characterized by a homogeneous distribution of physical and mechanical properties and stringent requirements to the raw materials used in production. The latest is essential in view of growing raw material deficit and shortage of large-sized wood raw material used for materials of solid wood. Its good physical and mechanical properties are due to the homogeneity of the material and the fact that MDF are composed of a plurality of lingo-cellulose components with high slenderness. This allows large active surface of the fibers and from there multiple contact areas within the boards which increasing quantities of both cohesion and adhesion bonds. Therefore this material finds numerous interior and exterior applications.

A major disadvantage of dry processed boards in comparisment of wet-processed ones is the existence of formaldehyde emissions.

There are successful attempts for production of eco-friendly MDF with the addition of enzyme lignin as a binder (Zouh et al. 2011; Mancera et al. 2011; Nasir et al. 2014). It should be emphasized that the use of enzyme lignin on an industrial scale is associated with a number of technological difficulties and lead to increased costs of the boards.

Calcium lignosulfonate is a residual product from the production of cellulose by the sulphate method. Lignosulfonates have a very low degree of harmfulness and find use, as a binder for the animal feed additives and in the production of pellets. They have relatively low glass point. A major advantage of lignosulfonates, compared with the enzyme lignin, is that they can be introduced into MDF in the form of solutions, to facilitate their even distribution and activation of the connections with the wood fibers.

Mechanical properties of MDF are essential in order to assess the suitability of the material for use in the furniture industry.

This defines the relevance of a study on the influence of the content of calcium lignosulfonate on the mechanical properties of MDF.

MATERIALS AND METHODS

The main goal of this study is to be determined the influence of the content of calcium lignosulfonate in the composition of MDF to their mechanical properties. To be fulfilled this goal in laboratory conditions they were produced MDF containing urea-formaldehyde resin of 5% and with variation in the content of calcium lignosulfonate from 0 to 20% with increment of 5%. The boards were produced with a density 850kg.m-3. For the production of MDF in laboratory conditions was used thermo-mechanical pulp of distributions in Bulgaria tree species – total content of beech (*Fagus silvatica* L) and cerris oak (*Quercuss cerris* L) of 80% and 20% content of poplar (*Populus alba* L). Wood fiber mass was with moisture content of 11%.

It was used calcium lignosulfonate with characteristic as follow: calcium – up to 6%; reduced sugars – up to 7%; dry content – 93%; acid factor in a 10% solution - pH = 4,3±0,8; bulk density – 550kg.m^{-3}.

In order to more uniform distribution and easier activation of the calcium lignosulfonate it is inserted into the pulp in the form of a solution having a concentration of 30%, Fig.1.

Fig. 1.
Calcium lignosulfonate.

The mechanical properties of MDF were determined by standard methods (EN 310; EN 316; EN 323; EN 622-5). For each property were used in eight test specimens per board. And main statistical parameters (average, standard deviation, probability) were calculated.

The data were processed by the methods of regression analyze and it was displayed approximating function to the influence of the content of calcium lignosulfonate on the MDF properties.

As a measure of accuracy of the regression models was used the coefficient of determination – R^2 (Trichkov 2015)

RESULTS AND DISCUSSION

The summarized results for the properties of MDF, with different participation of lignosulfonate are presented in Table. 1.

Table 1

Experimental results for physical and mechanical properties of MDF

Board №	**Content of calcium lignosulfonate *Px*, %**	**Density ρ, kg.m^{-3}**			**Bending strength (MOR), *fm*, N.mm^{-2}**			**Modulus of elasticity (MOE), *Em*, N.mm^{-2}**		
		Average	STDV	*P*-value	Average	STDV	*P*-value	Average	STDV	*P*-value
1	0	843.96	89.05	0.037	25.18	4.06	0.040	2867.50	412.41	0.036
2	5	845.98	44.97	0.019	30.63	3.56	0.029	3645.00	308.73	0.021

3	10	836.18	31.17	0.013	34.73	2.38	0.017	4253.75	427.18	0.025
4	15	839.19	68.46	0.029	27.16	2.91	0.027	4151.25	303.10	0.018
5	20	844.45	66.28	0.028	24.05	1.47	0.015	3445.00	182.13	0.013

Analysis of experimental results for bending strength of MDF

The dependence of bending strength of MDF from the content of lignosulfonate, at range of variation from 0 to 20%, is described by the equation of the regression of the form:

$$\hat{f}_m = 25.38 + 1{,}53.P_x - 0.08P_x^2, \text{ N.mm}^{-2}, \quad (1)$$

where: $\hat{f}_m$ is predicted value for bending strength, N.mm^{-2};

Px – content of calcium lignosulfonate in MDF, %.

The equation is characterized by the coefficient of determination R^2 = 0.82. Dependence is represented graphically in Fig.2.

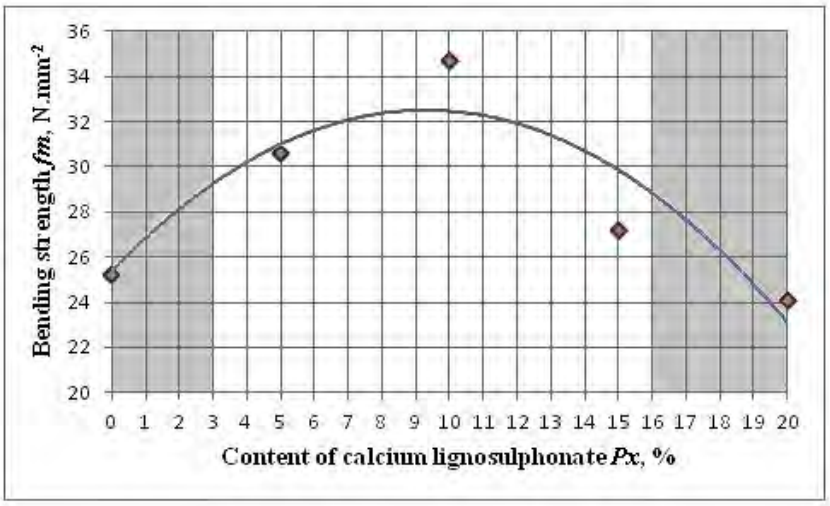

Fig. 2.
Variation of bending strength of MDF in dependence from the content of calcium lignosulfonate.

Under the conditions of the experiment bending strength varied from 24 to 34.7N.mm^{-2}. The highest bending strength was observed in MDF with 10% lignosulfonate. Bending strength increases with the increase in the content of lignosulfonate from 0% to 10%, and after passing 10%, which appears to be the inflection point, there is a decrease in value of that property. The lowest value of bending strength was accounted at MDF without lignosulfonate (0%) and with 20% of lignosulfonate, as the values of bought MDF are commensurate with differences within the statistical error. Whit the addition of up to 10% lignosulfonate the bending strength of MDF increases from 25 N.mm^{-2} at boards without lignosulfonate to 35N.mm^{-2}, or it is observed an improvement of the property by 1.4 times.

All boards meet the requirements in terms of bending strength for general purpose and for use in dry conditions.

In a study by the approximated function is established that MDF with a content of lignosulfonate from 3 to 16% meets the requirements of such intended for load-bearing boards (EN 622-5). The gray area in the figure is marked MDF with a content of lignosulfonate in which the boards do not meet those requirements.

Analysis of the experimental results for the modulus of elasticity of MDF

The dependence of the modulus of elasticity in bending of MDF from the content of lignosulfonate over a range of variation of 0 to 20%, is described by the equation of the regression (approximating function) of the type:

$$\hat{E}_m = 2814.70 + 243.44.P_x - 10.51.P_x^2, \text{ N.mm}^{-2}, \quad (2)$$

where: $\hat{E}_m$ is predicted value of modulus of elasticity, N.mm^{-2};

Px – content of calcium lignosulfonate in MDF, %.

The equation is characterized by the coefficient of determination R^2 = 0.98.

Figure 3 illustrates the variation of the modulus of elasticity of MDF in dependence of the content of lignosulfonate.

Under the conditions of the experiment, content of urea-formaldehyde resin of 6% and lignosulfonate content between 0 and 20%, the modulus of elasticity of MDF is changed in the interval from 2868 to 4254N.mm^{-2}. The lowest value of modulus of elasticity is accounted in MDF without lignosulfonate and the best values of the property in boards with 10% content of lignosulfonate. After raising the content of lignosulfonate of more than 10% the modulus of elasticity declined.

The deterioration in the boards by increasing the content of the lignosulfonate from 10% to 15% is not significant. While the transition from 15% to 20% content of lignosulfonate lead to significant decrease in the values of the modulus of elasticity.

The improvement in the values of the modulus of elasticity of MDF with the addition of 10% lignosulfonate as compared to those without lignosulfonate is 1.5 times.

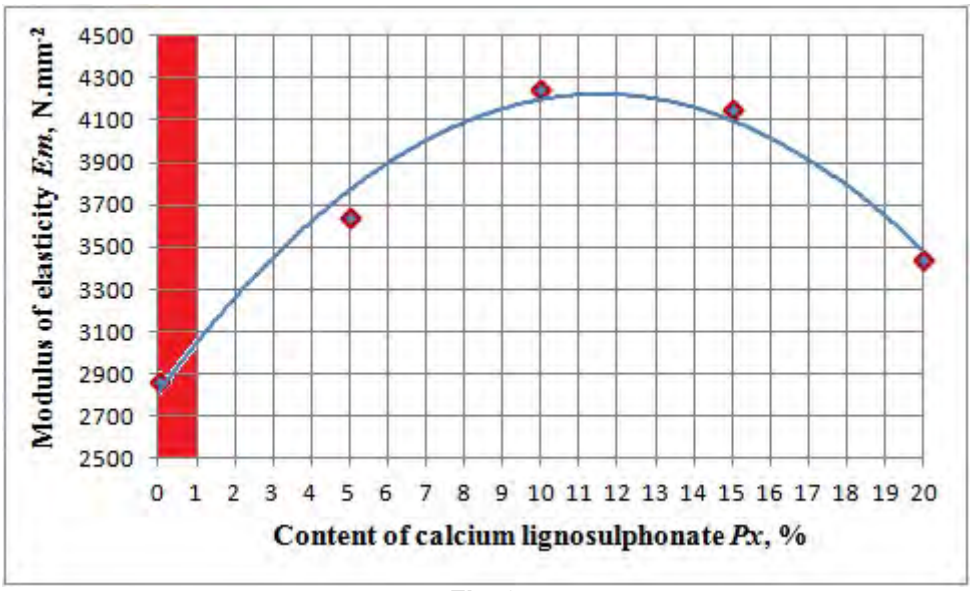

Fig. 3.
Variation of modulus of elasticity of MDF in dependence from the content of calcium lignosulfonate.

With respect to the property modulus of elasticity in bending, all boards meet the requirements for general purpose and use in humid conditions. The MDF containing lignosulfonate of one percent or more comply with the requirements for load-bearing boards and use in humid conditions (EN 622-5). With the red zone in Figure 3 are marked MDF with content of lignosulfonate in which the requirements for the modulus of elasticity for use as load-bearing boards are not achieved.

In tested mechanical properties of MDF, bending strength and modulus of elasticity in bending, there is a decrease after passing the lignosulfonate content of 10%. An explanation of these results can be given with the greater fragility of the boards when increasing the content of lignosulfonate and a high content of the steam-gas mixture in the process of pressing, associated with the increase in moisture content with an increased content of lignosulfonate solution. To be given more specific answer to this question it should be studied and other factors affecting the process as the influence of the dry content of lignosulfonate solution, influence of the hot pressing regimes, etc.

CONCLUSIONS

As a result of the conducted study on the influence of the participation of lignosulfonate in composition of MDF on its properties, can be made the following conclusions:

1) The addition of lignosulfonate up to ten percent in the composition of MDF significantly improves bending strength and modulus of elasticity in bending of boards;
2) MDF with ten percent lignosulfonate meet the highest classes of requirements for bending strength and modulus of elasticity in bending;
3) The relationship between the content of lignosulfonate and mechanical properties of the MDF is of the second degree, as it is observed inflection point in content of lignosulfonate by ten percent;

4) Upon addition of amounts more than ten percent lignosulfonate, under conditions of the experiment, is observed degradation, respectively decreasing, of bending strength and modulus of elasticity in bending of MDF.

In conclusion with the addition of lignosulfonate, which is a waste product in the production of cellulose, are achieved the most stringent requirements for mechanical properties of MDF. As the content of the urea-formaldehyde resin is only 5%, i.e. the boards are with a very low content of free formaldehyde.

ACKNOWLEDGEMENT

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UTILIZATION OF SILICA FUME IN MANUFACTURING OF CEMENT BONDED PARTICLEBOARDS

Husnu YEL
Artvin Coruh University, Faculty of Forestry,
Dept. of Forest Industrial Engineering, Artvin, Turkey
Tel: +90 466 2151000, Fax: +90 466 2151055, E-mail: yel33@artvin.edu.tr

Hulya KALAYCIOGLU
Karadeniz Technical University, Faculty of Forestry,
Dep. of Wood Industrial Engineering, Trabzon, Turkey
E-mail: khulya@ktu.edu.tr

Ugur ARAS
Karadenzi Technical University, Faculty of Forestry,
Dep. of Wood Industrial Engineering, Trabzon, Turkey
E-mail: uaras@ktu.edu.tr

Abstract

Silica fume, which is a highly reactive pozzolanic byproduct of producing silicon metal or ferrosilicon alloys, is known to improve both the mechanical characteristics and durability of concrete. This study investigates the effect of using silica fume as cement replacement materials on some properties of cement bonded particleboard from Douglas fir woods [Abies mordmanniana (Stev.) Spach. Subsp. Nordmanniana]. For this purpose, the three-layer cement bonded particleboards [CBPBs] with 1200 kg/m^3 target density and 1/2 - 1/3 wood-cement ratios were produced by replacing cement with different percentages of silica fume (10, 15, 20%). As cement curing accelerator, $CaCl_2$ was used at 5% ratio (based on cement weight) for all the boards. The CBPBs produced were tested, according to the related EN Standards, to determine their mechanical and physical properties including modulus of rupture, modules of elasticity, internal bond strength, screw withdrawal strength, density, moisture content, water absorption and thickness swelling. The modulus of rupture values ranged from 9 MPa to 13.6 MPa, while the modulus of elasticity values ranged from 5000 MPa to 6500 MPa. The internal bond strength values ranged from 1.05 MPa to 1.95 MPa. It was recorded that silica fume performed the improvements up to 20% at modulus of rupture and up to 40% at internal bond strength of the CBPBs. The mechanical properties of all the board types were over the requirements by EN 634-2 standard. Silica fume provided an improvement up to 50% at the thickness swelling values of the CBPBs. The results demonstrated that silica fume had a significant effect on the properties of CBPBs.

Key words: *cement bonded particleboards; nordmann fir; silica fume; technological properties.*

INTRODUCTION

Cement bonded particleboards (CBPBs) have been rapidly accepted and used in many countries for application in the building industry, because they possess a lot of outstanding merits including water, fire, decay, moisture and weather resistance, low cost, no health hazards, high stiffness, high durability and simple production processes etc., compared to conventional wood-based building materials (Wei et al. 2004; Maail et al. 2011). However, there are some problems impeding the development of the CBPBs. The most important one of them is that alkali-and water soluble sugars and extractive in wood inhibite the hydration reaction of cement, resulting in low strength value of wood-cement composites. Some methods such as NaOH, hot and cold water extractions, addition of effective chemicals, CO_2 Injection have been applied to improve the compatibility and to overcome this problem between wood and cement. Wood-cement ratio, water-cement ratio, cement type, addition type and amount, wood particle geometry and harvesting season, pressing conditions, in addition to wood species, also have influence on the quality of wood-cement composites.

Most researches have been focused on understanding the inhibitory properties of wood species, the compatibility of wood when mixed cement and water and improving the compatibility between wood and cement. However, because CBPBs is used mainly for building applications, it appears logical to pay more attention to the relation between the hydration process of wood-cement-water mixtures and the strength development of board (Wei et al. 2000).

Ordinary Portland cement is more expensive than wood materials. Therefore, it is a costly component and is responsible for a high proportion of the raw material cost (Wei et al. 2000). The application and use of mineral admixtures in concrete and wood-cement composites have been widely studied in recent decades to improve the resistance and durability of their composites and reduce the cement consumption (Kanning et al. 2014).

Silica fume is a byproduct resulting from the reduction of high – purity quartz with coal or coke and wood chips in an electric arc furnace during the production of silicon metal or silicon alloys. It is a material which may be a reason of air pollution. It is much cheaper than cement. Therefore, it is very important in terms of economical point of view (Rasol 2015). Hydration of cement primarily involves the reaction of calcium silicates C_3S and C_2S with water (H) to produce calcium silicate hydrate (C-S-H) and calcium hydroxide [$Ca(OH)_2$] (Young et al. 1974). The silica fume reacts with this $Ca(OH)_2$ in cement paste to form calcium silicate hydrate gel (C-S-H).

Silica fume is known to enhance both the mechanical characteristics and durability of concrete. Effects of silica fume on mechanical and durability of concrete were investigated by many researchers (Amudhavalli et al. 2012; Ajay et al. 2012; Ghutke and Bhandari 2014; Srivastava et al. 2014) and they reported the inclusion of silica fume up to 15% replacement level with cement in concrete have improved the bond strength, the bond strength of silica fume concrete increased in the range of 37 - 43% as compared to the referral concrete. In addition, it was reported that silica fume also decrease the voids, capillary, absorption and porosity of concrete because fine particles of silica fume reacts with lime present in cement.

Although many studies related to usage of silica fume as cement replacement materials in concrete have been carried out, there is no a comprehensive study about usage of silica fume as cement replacement materials in wood-cement composites and its effects on properties of the boards. Therefore, this study will be useful in terms of utilise of silica fume in wood-cement composite industry and thus, increasing its economic value

OBJECTIVE

The objectives of this study were to investigate the effect of silica fume on physical and mechanical properties of cement bonded particleboards from douglas fir woods, to determine the optimal silica fume usage ratio, to reduce its harmful effects to the environment and to increase its economic value by expanding the usage areas of silica fume. Therefore, the CBPB panels were produced using two wood-cement ratios (1/2 and 1/3) and three silica fume ratios of 10, 15 and 20% as a substitute for cement. The mechanical and physical properties, including modulus of rupture (MOR), modules of elasticity (MOE), internal bond strength (IB), screw withdrawal strength (SW), density (D), moisture content (MC), water absoption (WA) and thickness swelling (TS), of CBPBs, respectively, were determined according to the related standards.

MATERIAL, METHOD, EQUIPMENT

The Douglas fir woods [*Abies mordmanniana (Stev.) Spach. Subsp. Nordmanniana*] used in the this study were supplied as sawmill wastes by Trabzon Organized Industrial Zone, Turkey. Used as binder, ordinary Portland cement, CEM II B-M (P-LL) 32.5 R type was purchased from Askale Cement Co. in Turkey. In order to mitigate any adverse effects of water- and alkali-soluble products in wood, calcium chloride ($CaCl_2$), which purchased as solid state from Tetra Chemicals Europe AB in Sweden, was used as 5 wt. % based on weight of cement for all the board groups. Silica fume, supplied by Antalya Eti Elektrometalurji Co. in Turkey, was replaced with cement at ratios of 10, 15 and 20%.

Sawmill wastes were firstly chipped by means of a drum chipper and then grinded into smaller particles in a knife ring flaker. Then, the wood particles were classified in a laboratory type screen machine. The particles that remained between 3–1.5 mm sieve and 1.5–0.5 mm sieve were utilized in the core and outer layers, respectively. The shelling ratio (core layer/outer layers) was selected as 65/35% for all the board groups. The wood-cement ratios were 1/3 and 1/2, based on the oven dry weight for the three-layer CBPB manufacture. The amount of distilled water was adjusted by means of the formula below, founded by Simatumpang (1979):

$$Water\ (liter) = 0.35C + (0.30 - MC)W \quad (1)$$

where: C is the cement weight (kg), MC is moisture content (oven dry basis) of wood particles, and W is oven dry wood particle weight (kg). Experimental design is given in Table 1.

Table 1

Experimantel design

Board types	Silica fume (%)	Wood-cement ratio
A	0	1/3
B	10	1/3
C	15	1/3
D	20	1/3
A1	0	1/2
B1	10	1/2
C1	15	1/2
D1	20	1/2

The wood–cement-water mixes were mat formed as three layers on an aluminum plate inside a compact laminated mould. Hand-formed mats were compressed in a laboratory type hot press using a pressure of 18-20 kg/cm^3 for 24 hrs. In the first 8 hours of the pressing process, a temperature of 60°C was applied and then pressing was continued for 16 hrs in ambient temperature. All the boards were produced at a dimension of 450x450x10 mm and a target density of 1200 kg/m^3. To complete hydration of cement, the boards after pressing were conditioned in a climate room with a temperature of 20°C and a relative humidity of 65% for 28 days and then cut into test samples according to the requirements stated in the European Standards.

Physical and mechanical properties including density, moisture content, water absorption, thickness swelling, modulus of rupture, modulus of elasticity, internal bond strength and screw withdrawal strength, of the boards were determined according to EN 323 (1993), EN 322 (1993), ASTM D1037 (2006), EN 317 (1993), EN 310 (1993), EN 319 (1993) and EN 320 (2011), repectively.

RESULTS AND DISCUSSION

Mechanical Properties

The averaged measured values of MOR and MOE were plotted against wood and silica fume amounts in Fig. 1. It can be observed that MOR values were improved with increasing the wood content (wood/cement ratio from 1/3 to 1/2) in the boards in contrast to MOE. Papadopoulos et al. (2006) and Ashori et al. (2012) obtained the similar results. An increase in the amount of wood up to a certain amount enhanced the compression factor in the boards due to that wood is lighter than cement and this led to increase the contact surface area between the particles and the distribution of load applied on the boards. This resulted in more strength of the boards. Moslemi and Pfister (1987) explained the stuation with smilar expressions. However, MOE values decreased with increasing wood content due to fact that wood has less elasticity modulus than concrete.

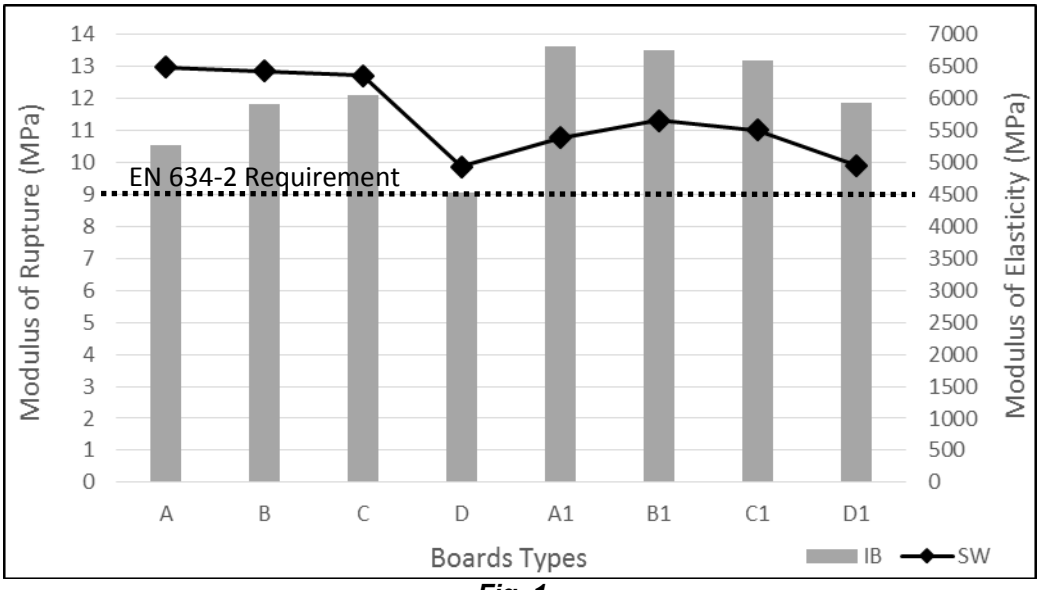
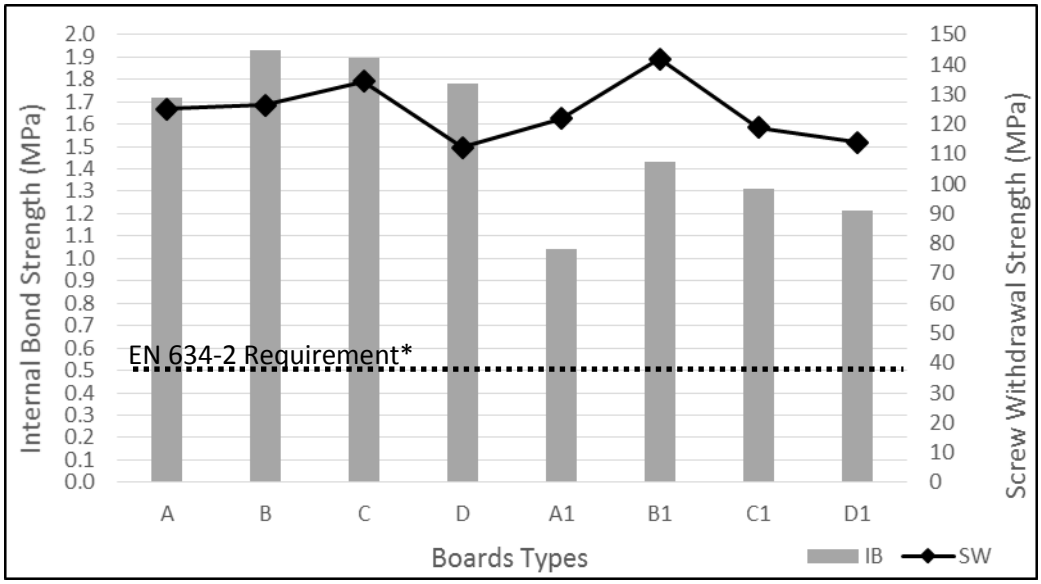


Fig. 1.
MOR and MOE properties of the boards.

MOR and MOE values of all the boards met the requirements for ordinary Portland cement bonded particleboards for use in dry, humid and external conditions stated in EN 634-2 (2007). The highest values were obtained from the boards with 15% silica fume among the boards with 1/3 wood-cement ratios.The figure showed that adding silica fume up to 15% significantly improved MOR values of the boards with 1/3 wood-cement ratios and MOE values of the boards with 1/2 wood-cement ratios.

The averaged measured values of IB and SW were given in Fig. 2. Contrary to MOR values, IB values decreased with increasing wood content. The highest IB and SW values were obtained from the B and B1 groups including %10 silica fume while the lowest values were obtained from the referance boards (A and A1) including no silica fume. IB values of all the boards were over the minimum values required for ordinary Portland cement bonded particleboards for use in dry, humid and external conditions stated in EN 634-2 (2007).



*This standard requirement is valid only for IB values

Fig. 2.
Properties of IB and SW strength of the boards.

Hydration of cement produces calcium hydroxide [Ca(OH)2] and calcium silicate hydrate [C–S–H gel]. C-S-H gel is the main product of the hydration of Portland cement and is primarily responsible for the strength in cement based materials. $Ca(OH)_2$ is is a by-product which is unresistant, water-soluble and non-contributing to the strength of cement based products. Silica fume reacts with $Ca(OH)_2$ and generates C-S-H gel (Metha 1987). This leads to an increase in the strength of cement bonded particleboards. The reason of the improvement in mechanical properties of the boards with silica fume may be that the amount of calcium silicate hydrate in the boards increased due to fact that silica fume reacted with $Ca(OH)_2$.

Silica fume, wood-cement ratio and interaction of both had a significant effect on mechanical properties (MOR, MOE, IB and SW) based on the results of statistical analysis at 99% confidence level. The results of homogenous subsets of the board were given in Table 2.

Table 2

Homogenous subsets for mechanical properties of WCPB

Board Types	MOR	MOE	IB	SW	Board Types	MOR	MOE	IB	SW
A	c	a	b	b	**A1**	a	c	d	b
B	b	b	a	b	**B1**	b	a	a	a
C	a	c	a	a	**C1**	c	b	b	bc
D	d	d	b	c	**D1**	d	d	c	c

Physical Properties

Thickness swelling and water absorption in wood-cement composites mainly depend on wood-cement ratio and hydration reaction of cement. Figure 3 showed the averaged thisckness swelling values of the boards soaked for 2 and 24 hours. The boards with 1/3 wood-cement ratio had much lower thickness swelling values than that of tha boards with1/2 wood cement ratio. In additon, it was observed that silica fume significantly improved the thickness swelling values of the boards. All the boards with 1/3 wood-cement ratio met the requirements (<1.5%) stated in EN 624-2 (2007).

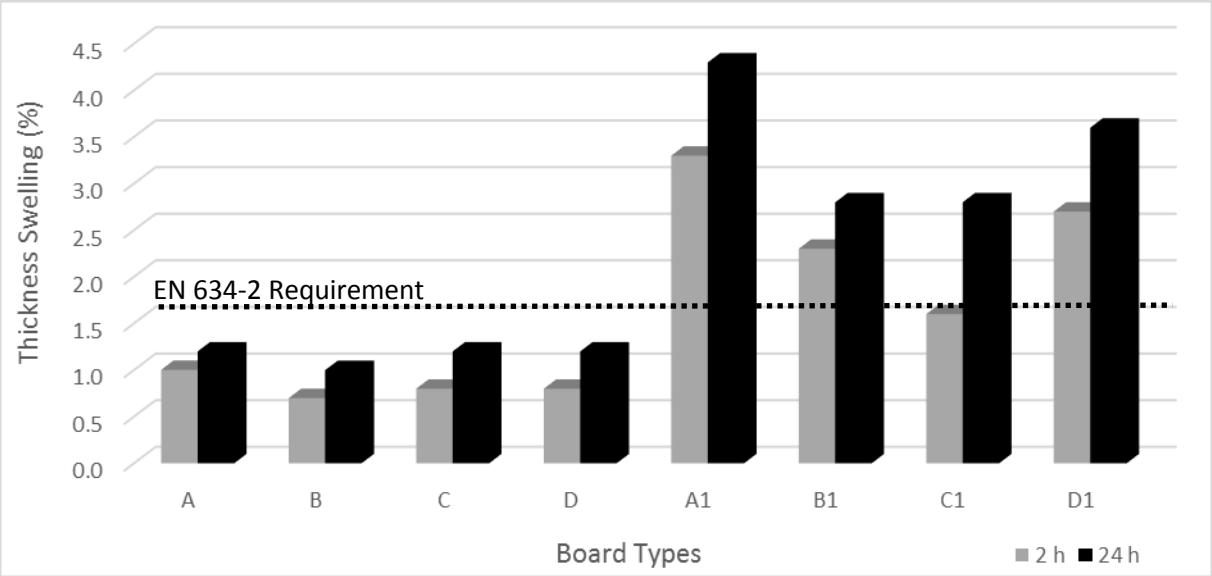


Fig. 3.
Thickness swelling values of the boards.

Physical properties of the boards including density, mositure content, water absorption and thickness swelling were given in Table 3. Moisture content (ranged from 9.4% to 11.9%) of all the boards met the requirement (MC: 9-12%) stated in EN 634-1 (2007). Using silica fume up to 15% in manufacturing of the boards decreased WA and TS values of the boards. Particle size of silica fume is approximately 100 time smaller than that of Portland cement (Xu and Chung 2000). Therefore, silica fume may have created a tighter structure by filling the voids in the board. This may be the reason why the boards containing silica fume had the less TS and WA values. In addition, the increased hydration products (C-S-H) and the improved strength values also may have led to the lesser WA and TS values of the boards than the others.

Table 3

Physical properties of the boards

Board Types	Density (kg/m^3)	Moisture Content(%)	Water Absorption (%)		Thickness Swelling (%)	
			2 h	24 h	2 h	24 h
A	1.28	9.4	10.1 c	14.2 c	1.0 c	1.2 b
B	1.27	11.1	8.7 b	12.9 b	0.7 a	1.0 a
C	1.19	11.9	7.2 a	11.8 a	0.8 b	1.2 b
D	1.20	10.3	14.3 d	17.8 d	0.8 b	1.2 b
A1	1.22	9.7	15.8 c	20.4 b	3.3 d	4.3 c
B1	1.14	11	13.1 b	18.9 a	2.3 b	2.8 a
C1	1.17	11	11.1 a	18.9 a	1.6 a	2.8 a
D1	1.13	10.4	16.1 d	21.3 c	2.7 c	3.6 b

It was founded that the boards with 1/2 wood-cement ratio had more WA ratio than that of the boards with 1/3 wood-cement ratio because the wood content increased. Zhou and Kamdem (2002), Ashori et al. (2012) and Sudin and Swamy (2006) reported the similar results. Homogenity subsets for WA and TS values of the boards were also shown in Table 3. According to the results of statistical analysis (p:0.0001), silica fume and wood-cement ratio and interaction of both had a significant effect on WA and TS values of the boards.

CONCLUSIONS

The results obtained in this study investigated the effect of silica fume on physical and mechanical properties of cement bonded particleboards from Douglas fir woods, demonstrated that using silica fume up to 15% in manufacturing of wood-cement composites significantly improved their physical and mechanical properties. In addition, it was observed that silica fume had more effect on the boards with 1/3 wood-cement ratio than the boards with 1/2 wood-cement ratio. Using silica fume as cement replacement in manufacturing of wood cement composites will provide: 1) producing high strength and durability of wood-cement composites, 2) increasing the econamical value of silica fume, 3) decreasing the cost of wood-cement composites because silica fume is much cheaper than cement and 4) decreasing the air pollution caused by silica fume.

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UTILIZATION OF HAZELNUT SHELLS AS FILLER IN LDPE/PP BASED POLYMER COMPOSITES

Kadir KARAKUS
Kahramanmaras Sutcu Imam University
Faculty of Forestry, Department of Forest Industry Engineering
Address: KSU Avsar Yerleskesi. 46100 Kahramanmaras, Turkey
E-mail: karakus@ksu.edu.tr

İbrahim Halil BASBOGA
Kahramanmaras Sutcu Imam University
Faculty of Forestry, Department of Forest Industry Engineering
Address: KSU Avsar Yerleskesi. 46100 Kahramanmaras, Turkey
E-mail: ihbasboga@ksu.edu.tr

İlkay ATAR
Kahramanmaras Sutcu Imam University
Faculty of Forestry, Department of Forest Industry Engineering
Address: KSU Avsar Yerleskesi. 46100 Kahramanmaras, Turkey
E-mail: iatar@ksu.edu.tr

Fatih MENGELOGLU
Kahramanmaras Sutcu Imam University
Graduate School of Natural and Applied Science, Materials Science and Engineering
Faculty of Forestry, Department of Forest Industry Engineering
Address: KSU Avsar Yerleskesi. 46100 Kahramanmaras, Turkey
E-mail: fmengelo@ksu.edu.tr

Abstract

Polymer based composites manufactured using thermoplastic and lignocellulosic materials are increased recently. Hazelnut shells as lignocellulosic were considered as a potential filler for polymer composites. This study measured the effect of filler loading on the mechanical properties of low density polyethylene (LDPE) and polypropylene (PP) based polymer composites. PP and LDPE were used as thermoplastic polymer. Hazelnut shells flours (HSF) were used as filler. The blends of PP, LDPE and HSF were compounded using single screw extruder and test samples were prepared through injection molding. The tensile, flexural and impact properties of the produced composites were determined in accordance with ASTM D638, ASTM D790, and ASTM D256, respectively. As a result, with increase of filler loading for PP, tensile strength (TS), elongation at break (EatB) and flexural strength (FS) were decreased while tensile modulus (TM) and flexural modulus (FM) were increased. Impact strength (IS) of PP based polymer composites were not changed. Also the addition of filler loading into LDPE matrix reduced TS, EatB, and IS while FS, FM and TM were increased. In this study, manufactured all composites provided flexural properties for the plastic lumber applications by *the ASTM D 6662 (2001) standard.*

Key words: *thermoplastic composite; low density polyethylene; polypropylene; hazelnut shells; mechanical properties.*

INTRODUCTION

Polymer composites can be consisted thermoplastic polymers such as polyethylene (PE), polypropylene (PP), polyvinyl chloride (PVC), polystyrene (PS) and lignocellulosic fillers such as wood flours (pine, poplar, beech, eucalyptus, etc.) and agricultural wastes (wheat straw, rice husk, hemp, kenaf fibers, walnut, hazelnut shells, tea mill waste, etc.). Its low density, low cost, their availability, renewability, high specific properties, low hygroscopicity, high dimensional stability, eco-friendliness and ease of processing can be listed among the advantages of the polymer based composites filled by lignocellulosic materials (Dobreva et al. 2006; Mengeloğlu and Karaku, 2008; Kaymakci and Ayrılmıs 2014; Donmez Cavdar et al. 2015). Polymer based composites have wide application areas (decks, walkways, automobile industry, house hold applications, fence, door and window frames, music and

sports materials, etc.) due to the their advantages over both the plastic and wood material (Yang et al. 2004; Muhammed et al. 2015).

The use of fillers in polymer based composites as published in the literature is also summarized. Several studies were conducted to investigate the use of lignocellusic fillers in polymer based composites. Bamboo was also used as filler in thermoplastic (PLA and PBS) matrix and mechanical properties were compared (Lee and Wang 2006). Hamid et al. investigated the physical and mechanical properties of high density polyethylene (HDPE) based reinforced with rice husks and sawdust. Reddy and Yang developed polymer based on mechanical and physical properties of soyprotein-jute and PP-jute composites. There are also some studies on the thermoplastic and lignocellulosic materials. Zhou et al. investigated the mechanical properties of the composites made from ramie filled poly (lactic acid).

There is tremendous amount of forest products industry and agricultural wastes available in Turkey and they are not utilized rationally. It is possible to utilize these wastes in the manufacture of polymeric composites (Mengeloğlu ve Karakus 2008). Nutshells are renewable lignocellulosic materials that can be obtained as agricultural byproducts. They are often utilized in relatively low-value applications such as composts, mulches, fertilizers, animal feed, burned or left in the agricultural land after harvest (Sutivisedsak ve ark. 2012). Annual hazelnut amount is around 768.300 tons per year between 2009-2014. Annual hazelnut husk amount is around 400,000 tons. Turkey is largest hazelnut producer with a supply of 70% followed by Italy, USA and Spain in the world. Nutshell as a filler consist of 25% to 30% cellulose and hemicelluloses and 30% to 40% lignin (Sarıca and Cam 2000; Tuik 2010; Salasinska and Ryszkowska 2012; Avci et al. 2013; Boran 2016).

The aim of this study is to investigate the usability of hazelnut shell flour as fillers in polymer based composites. Also, this study measured the effect of filler loading on the mechanical properties of LDPE/PP based polymer composites. The mechanical and physical properties of the polymer composites produced from hazelnut shell flours and LDPE/PP were determined.

EXPERIMENTAL

Materials

Low-density polyethylene (LDPE) and polypropylene (PP) were used as thermoplastic matrix. These plastic were used as received from the manufacturer. Hazelnut shell flours (HSF) were used as lignocellulosic filler. The fillers were collected from Trabzon, Turkey. Zink borate and paraffin wax (K.130.1000) was used as a lubricant.

Polymer composite manufacturing

The hazelnut shell wastes granulated in Wiley Mill into the flour form. These flours, screened and retained on 60 mesh-size screen (0.25mm), were used in this study. The classified fillers were dried in oven at 103°C (±2) for 24 hours. The experimental design of the study was presented Table 1. Depending on the formulation given LDPE or PP, and HSF and paraffin wax were dry-mixed in a high-intensity mixer to produce a homogeneous blend. These blends were compounded in a single-screw extruder at 40rpm screw speed in the temperatures (barrel to die) of 170-180-185-190-200°C. Extruded samples were cooled in water pool and then granulated into pellets. The pellets were dried in oven at 103°C (±2) for 24 hours. The pellets were injection molded into tensile, flexural test samples using an HDX-88 injection molding machine at a barrel temperature of between 180°C and 200°C (injection pressure: 100 bar, injection speed: 80mm/sec., screw speed: 40rpm., cooling time about 30s.).

Table 1

Manufacturing schedule of composites (%)

Group ID	Polymer Type (PP) / (PE)	Polymer (%)	Hazelnut shell flours (%)	Zinc borate (%)	Wax (%)
PE-0	PE	96.0	0.0	2	2
PE-1	PE	78.5	17.5	2	2
PE-2	PE	61.0	35.0	2	2
PE-3	PE	43.5	52.5	2	2
PP-0	PP	96.0	0.0	2	2
PP-1	PP	78.5	17.5	2	2
PP-2	PP	61.0	35.0	2	2
PP-3	PP	43.5	52.5	2	2

Polymer composite testing

Testing of the samples was conducted in a climate-controlled testing laboratory. Densities were measured by a water displacement technique according to the ASTM D 792 standard. Tensile, flexural and impact properties of all samples were determined according to ASTM D 790, ASTM D 638, and ASTM D 256, respectively. The span length of each specimen was 80mm, with the rest left as overhang for flexural testing. The rate of crosshead motion was 2.0mm/min, which is calculated according to the ASTM standard. Tests were performed at a rate of 5.0mm/min. Dog-bone shape samples were used (Type III) for tensile testing. Ten samples for each group were tested. Flexural and tensile testing were performed on Zwick 10KN while a HIT5,5P by Zwick™ was used for impact property testing on notched samples. The notches were added using a Polytest notching cutter by RayRan™.

Data analysis

Design-Expert® Version 7,0,3 statistical software program was used for statistical analysis.

RESULTS AND DISCUSSION

LDPE based or PP based polymer composites were produced in the density range of 0,91-1.50g/cm^3. Mean density values are presented in Table 2. Plastic type and filler amounts were statistically significant. LDPE based composites had higher density values compared to PP based ones due to the density differences of base polymers. Hazelnut shell flours filled composites provided slightly higher density values compared to neat polymers for both LDPE and PP. In addition, density of the composites was increased with filler loading.

Table 2

Density of the manufactured polymer composites

Group ID	Density (g/cm^3)
PE-0	0.96 (0.011)
PE-1	1.27 (0.04)
PE-2	1.36 (0.02)
PE-3	1.50 (0.03)
PP-0	0.91 (0.005)
PP-1	1.20 (0.03)
PP-2	1.27 (0.009)
PP-3	1.38 (0.03)

* Values in parenthesis are standard deviations.

In this study, tensile, flexural and impact properties of all samples were determined. Mechanical properties of the polymer composites produced with HSF were summarized in Table 3. The arithmetic mean and standard deviation values were given for each group in the table. Two different polymers and three different ratios of filler material (hazelnut shell flours) were used.

Table 3

Mechanical properties of polymer based composites

Group ID	Tensile Strength (MPa)	Tensile Modulus (MPa)	Elongation at Break (%)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Impact Strength (J/m)
PE-0	9.04 (0.2)*	81.02 (5.72)	103.75 (11.76)	6.07 (0.13)	133.82 (166.13)	436.45 (39.01)
PE-1	8.08 (0.06)	151.51 (23.63)	30.50 (7.55)	10.36 (2.47)	1301.10 (102.82)	135.55 (12.78)
PE-2	6.14 (0.16)	186.06 (21.24)	11.09 (2.30)	10.30 (0.27)	447.18 (41.79)	62.15 (5.94)
PE-3	3.68 (0.45)	263.69 (28.27)	4.68 (0.09)	9.69 (0.18)	786.03 (32.46)	29.97 (3.15)
PP-0	29.01 (0.94)	406.43 (49.25)	465.00 (52.85)	39.52 (1.65)	1077.18 (63.91)	15.21 (2.26)
PP-1	21.58 (0.54)	631.99 (17.69)	8.03 (0.88)	34.02 (1.13)	1307.18 (74.73)	18.85 (3.29)
PP-2	16.27 (0.48)	667.21 (14.37)	5.01 (0.52)	29.89 (1.37)	1660.29 (105.06)	18.22 (1.61)
PP-3	11.52 (0.34)	671.45 (34.26)	3.25 (0.39)	25.02 (0.65)	1917.68 (49.26)	18.22 (2.25)

* Values in parenthesis are standard deviations.

Tensile properties include tensile strength, tensile modulus and elongation at break. With the increasing of HSF loading tensile strength was significantly reduced in both LDPE and PP based composites. It is thought that the most important reason of decline is lack of bonding between polymer matrix and lignocellulosic fillers (Mengeloglu and Karakus 2008; Donmez et al. 2015). The polymer type was also effective on the tensile strength of the composite material. PP composites relatively provide better tensile strength compared to the LDPE composites. To mention of tensile modulus, rise of filler loading significantly increased the tensile modulus for both composites. Similar results for other wood flours filled polymer composites were also reported (Lee and Wang 2006). The filler has significant effect on elongation at break values for both composites. Significant reduction by addition of filler in elongation at break values was determined for both polymer matrixes.

Flexural properties include flexural strength and flexural modulus. The results showed that the flexural strengths are significantly affected by filler loading. Similar results were also reported for the flexural strength of other wood flour filled thermoplastic composites (Donmez Cavdar et al. 2015; Boran 2016). With filler loading flexural strength was reduced for PP based composites. PP composites relatively provide better flexural strength compared to the LDPE composites. The results of analysis show that the rate of HSF was effective on flexural strength for PP composites. Compared to control samples, while there is a reduction on the flexural strength for PP based composites, the increase has been observed on flexural strength for LDPE based composites. The polymer type was also effective on the flexural strength of the composite material.

With the increase of filler loading increased the flexural modulus for LDPE and PP based composites. Lignocellulosic fillers and polymers have different modulus of elasticity from each other. Lignocellulosic fillers have higher modulus of elasticity than polymer. This is caused to have better flexural modulus for composite from pure polymer. Therefore, flexural modulus increased with the rise of lignocellulosic filler loading. Addition of the lignocellulosic filler improves tensile modulus of the thermoplastic composites usually could simply be explained by the rule of mixtures (Matuana et al. 1998; Mengeloğlu and Karakus 2008). PP composites relatively provide better flexural modulus compared to the LDPE composites. In this study, produced polymer composite materials were usually considered as an alternative to the polyolefin-based plastic lumber decking boards. For polyolefin-based plastic lumber decking boards, ASTM D 6662 (2001) standard requires the minimum flexural strength of 6.9MPa. All composites produced in this study provided flexural strength values (9.69-39.52MPa) that are well over the requirement by the standard. ASTM D 6662 (2001) standard requires the minimum flexural modulus of 340MPa for polyolefin-based plastic lumber decking boards. All composites produced in this study provided flexural modulus values (447-1917MPa) well over required standards.

The results show that pure LDPE has higher impact. Impact strength reduced hazelnut shell flour was added to polymer matrix. There is little difference between impact strength of PP based. With the increase of filler loading decreased the impact strength for LDPE. This usually arises from increasing of brittleness of the composite material (Matuana et al. 1998; Mengeloglu and Karakus 2008).

CONCLUSIONS

Low density polyethylene (LDPE) and polypropylene (PP) based polymer composites including different ratios of hazelnut shell flour are manufactured by injection moulding. The mechanical properties of the produced polymer composite (tensile strength, tensile modulus, flexural strength, flexural modulus, elongation at break and impact strength) were determined.

Hazelnut shell flours (HSF) flour filled polymer composites were successfully produced and the following conclusions were reached:

1. PP based composites provided better mechanical properties compared to LDPE based composites.
2. Addition of HSF flour into polymeric matrices improved modulus values while reducing strength, elongation and impact values.
3. LDPE and PP based polymer composites provide adequate mechanical properties according to ASTM D 6662 (2001). As a result, HSF flour might be utilized as filler for LDPE and PP based polymer composites.

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COUPLING AGENT EFFECT ON THE PROPERTIES OF THERMOPLASTIC COMPOSITES FILLED SAND-DUST FROM MEDIUM DENSITY FIBERBOARD

Ayfer DONMEZ CAVDAR
Karadeniz Technical University, Faculty of Architecture
61080 Trabzon, Turkey
Tel: 0090 462 3771682, Fax: 0090 462 3255588, E-mail: adonmez@ktu.edu.tr

Hülya KALAYCIOGLU
Karadeniz Technical University, Faculty of Forestry
61080 Trabzon, Turkey
E-mail: khulya@ktu.edu.tr

Fatih MENGELOGLU
Kahramanmaras Sutcuimam University, , Faculty of Forestry
46100 Kahramanmaras, Turkey
E-mail: fmengelo@ksu.edu.tr

Abstract

The study investigates coupling agent effect on some properties of thermoplastic composites filled with sand dust from medium density fiberboard (MDF). The physical, mechanical and morphological properties of the composites produced with the extrusion method were accomplished by using sand dusts of medium density fiberboard (SD_MDF) as lignocellulosic material, high density polyethylene (HDPE) and polypropylene (PP) as thermoplastic polymer and maleic anhydride grafted HDPE and maleic anhydride grafted PP as coupling agents. Coupling agents at different ratios were used to improve the adhesion between fiber and polymers in production, and their effects on the properties were evaluated. Thickness swelling and water absorption of the samples decreased when used coupling agent. Except for impact strength, flexural and tensile properties of the composites which increased with presence of a coupling agent. In the light of obtained results, it was specified that use of coupling agent improved physical, mechanical and morphological properties of SD_MDF filled thermoplastic composites. In addition, optimum coupling agent usage rate were determined as 3%.

Key words: *coupling agent; thermoplastic composite; sand dusts of Medium Density Fiberboard; mechanical properties; physical properties.*

INTRODUCTION

Natural fibers play an important role as filler or reinforcer for polymer composite industry due to having low cost with low density and high specific properties (Boran 2016, Cavdar and Boran, 2016). Wood plastic composites are among the major natural fiber composites with a large production capacity.

The natural fiber filled thermoplastic composite production is not a simple and problem-free production. The most important problems in the production with the addition of natural fibers or flours into the plastic matrix can be summarized in two main categories. The first is moisture affinity and temperature sensitivity of natural fiber such as wood fiber/flour and the second is bonding problems due to the incompatibility between the natural fiber and the thermoplastic polymer. Coupling agents play a bridge between two materials by forming a chemical bonding with hydrophilic lignocellulosic material and increasing surface wettability of hydrophobic polymer chain (Oksman and Lindberg 1998, Zhang *et al.* 2002; Yang *et al.* 2007).

Much research has been done on the problem of incompatibility between wood flour and plastic and it has become necessary to transform wood flour surfaces into a hydrophobic structure through modification (Pritchard 1998, Cavdar *et al.* 2014). Various coupling agents have been used for this purpose. Among these agents, the most commonly used chemicals are synthetic polymers treated with maleic anhydride. In many studies on the use of compatibilizers, maleic anhydride grafted with synthetic polymers has proved to serve as a bridge between the lignocellulosic filler and the polymer matrix (Sanadi *et al.* 1997, Lu *et al.* 2000, San *et al.* 2008). The molecular weight and amount of the grafted maleic anhydride are important parameters for the effect of the compatibility. Maleic anhydride in maleic anhydride-grafted- polypropylene / polyethylene (MAPP / MAPE) provides a polar interaction

such as acid-base interaction and can covalently linked to hydroxyl groups on lignocellulosic fillers (Felix *et al.*1993, Sanadi *et al.*1995, San *et al.* 2008).

In studies aiming to determine optimum usage ratio of compatibilizer, it has recommended 2-8% and 1-4% of the weight of the wood for WPCs produced at 50:50 and 70:30 wood / plastic ratio, respectively. In some studies, it has been concluded that the use of 1-3% compatibilizer in relation to the total weight of the WPCs is appropriate (Maldas *et al.* 1989, Krzysik *et al.* 1990, Myers *et al.* 1993, Lu *et al.* 2000).

The concentration of the coupling agent determines the compatibility between the materials in the composite. Some researchers have reported that coupling agent at low concentrations improves mechanical properties of the composites while the mechanical properties decreased with using higher concentration. Therefore, optimum usage ratio for coupling agent should be taken into consideration in production of natural fiber filled thermoplastic composites.

OBJECTIVE

The main objective of the present research investigates the effect of various coupling agent usage ratios on physical, mechanical and morphological properties of thermoplastic composites filled with sand dusts from MDF.

MATERIALS AND METHODS

In this study, sand dusts of MDF panels were provided by Kastamonu Integrated Forestry Industry and Trade Inc., Turkey. The dusts (≈200 mesh) were generated from the sanding of the panels produced from beech, pines and oaks fibers and urea formaldehyde resin based on 65 % solid content. High density polyethylene (HDPE)(MFI/190°C/2.16 kg = 0.35 g/10 min) and polypropylene (PP) (MFI/230°C/2.16 kg = 5 g/10 min) were purchased by Petkim Petrochemical Co., Turkey. Coupling agents, maleic anhydride grafted polyethylene (MAPE) (Licocene PE MA 4351, density: 0.99 g/cm^3) and maleic anhydride grafted polypropylene (MAPP) (Licomont AR 504, density: 0.91 g/cm^3) were supplied by Clariant International Ltd, in Germany.

Composite Manufacturing

SD_MDF, coupling agent and wax with HDPE or PP were mixed with high-speed mixer (Teknomatik, Turkey), speed range 5–1000 rpm, for 5 min. The compounding was accomplished using a laboratory scale single screw extruder (Teknomatik, Turkey). The set temperatures of the extruder were controlled at 170°C, 175°C, 180°C, 185°C, and 190°C for five heating zones and the extruder screw speed was set to 40 rpm. The cooled pellets in water were granulated and dried at 105 °C for 24 h. The granulated and oven-dried pellets were pressed under 100 kg/cm^2 pressure, at 200°C temperature for 20 min using a mould dimension of 20cm by 20cm by 0.5cm. After pressing, the composites were conditioned in a climatic room with the temperature of 20 °C and the relative humidity of 65%. The raw material formulations used for the composites are presented Table 1.

Table 1

Parameters of manufacturing the thermoplastic composites

Composite panel type	Plastic type	Coupling agent	Composite formulations (%)			
			SD_MDF	Plastic	Coupling agent	Wax
Control_HDPE	HDPE	MAPE	40	54	-	3
Control-PP	PP	MAPP	40	54	-	3
A1	HDPE	MAPE	40	53	1	3
B1	HDPE	MAPE	40	51	3	3
C1	HDPE	MAPE	40	49	5	3
A2	PP	MAPP	40	53	1	3
B2	PP	MAPP	40	51	3	3
C2	PP	MAPP	40	49	5	3

Physical properties

Water absorption (WA) and thickness swelling (TS) of the thermoplastic composites were evaluated according to EN 317. Five samples for each group with dimensions of 50mm*50mm*5 mm, were used to determine the WA and TS tests. The conditioned samples were weighed and their thicknesses were measured, then the samples were dipped into water for 6 months. The weight and

thickness of the samples were measured periodically during six months. The samples were weighed, and their thicknesses were measured to calculate WA and TS rates.

Mechanical properties

Flexural and tensile properties and impact strength of the thermoplastic composites filled with SD_MDF were determined by ASTM D 638, ASTM D 790 and ASTM D 206, respectively. The flexural and tensile properties of all group samples were determined on Zwick Testing Unit with a capacity 10 kN (1000 kg). Impact strength samples were notched with a Polytest notching cutter by RayRanTM and tested on a Zwick HIT5.5P impact-testing machine.

Scanning Electron Microscope

Morphological properties of the composites were accomplished using A JEOL JSM-5500 scanning electron microscope (JEOL Ltd., Japan). The impact samples were cryogenically fractured in liquid nitrogen, and then were gold coated by sputtering technique before the microscopic observations. Images were taken at 50 X SEM micrograph magnifications.

RESULTS AND DISCUSSION

Thickness Swelling and Water Absorption

The results of TS and WA of SD_MDF filled thermoplastic composites with coupling agent are shown in Figures 1 and 2. The TS and WA values of all samples increased with increment of water immersion time. For the HDPE based composites, TS values of HDPE based composites were ranged between 0.41% and 1.70% while those of PP composites were ranged between 1.50% and 1.82% after six months immersion time in water. Regarding WA values were found as 3.97% and 5.86% after six months immersion time for HDPE based composites. These values for PP based composites were found as 7.90% and 8.83% after the six months.

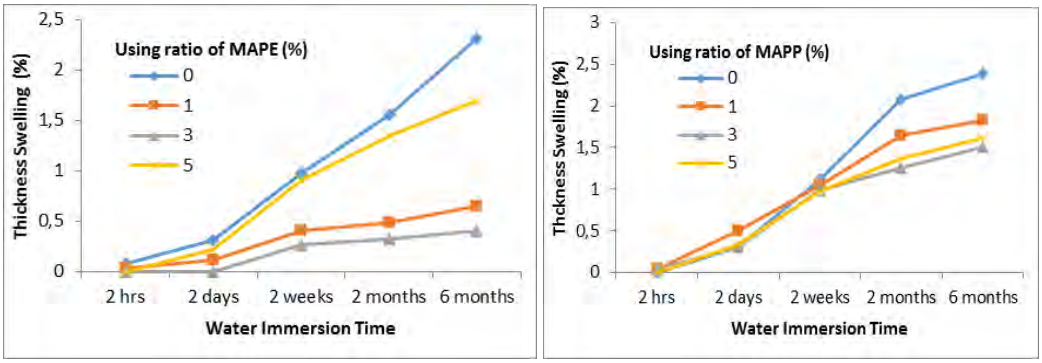


Fig. 1.
Coupling agent effect on thickness swelling of the thermoplastic composites.

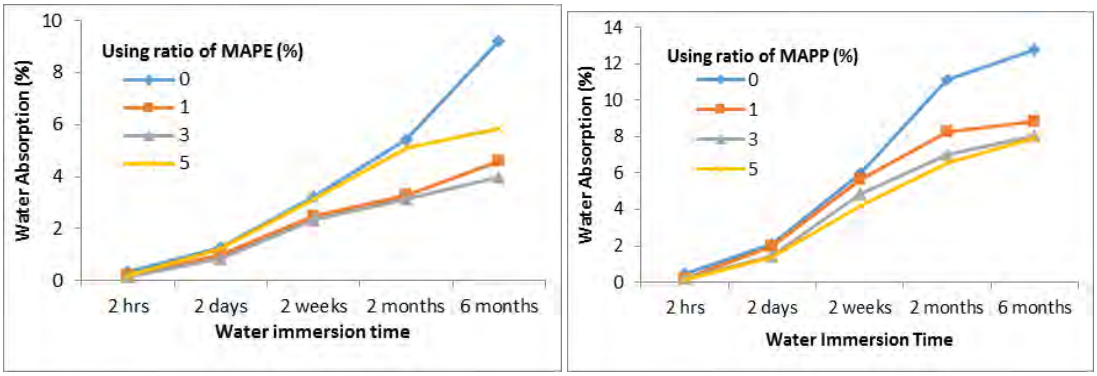


Fig. 2.
Coupling agent effect on water absorption of the thermoplastic composites.

The water absorption and thickness swelling of the composite samples were reduced with use of coupling agents. It has been supported by some reserachers that use of coupling agent decreased WA and TS of thermoplastic composites (Matuana *et al.* 2001, Panthapulakkal *et al.* 2005, Shakeri and Ghasemian 2010, Yang *et al.* 2005, Rozman *et al.* 2010, Hamzeh *et al.* 2011). This is probably due to the fact that the maleic anhydride in MAPE/MAPP reacts with the free -OH groups in the

lignocellulosic materials, forming the ester bonds. Therefore, amount of free -OH groups are reduced and the samples with coupling agent are less water uptake than the control samples. Another possible reason is that coupling agent may increase the crystallinity of the composite. Some researchers have concluded that, the crystallinity of the composites produced using the coupling agent was higher than that of the blends without using the coupling agent. The rate of water absorption would be lower in composites with coupling agent, depending on the reduced permeability in crystalline regions (Ichazo *et al.* 2001, Shakeri and Ghasemian 2010).

However, HDPE based composites had higher dimensional stability (lower WA and TS) than PP based composites. This may happen due to the different characteristics between these plastics (Cavdar *et al.* 2011)

Mechanical properties

The results of mechanical properties of thermoplastic composites filled with SD_MDF and coupling agent are shown in Table 2.

Table 2

Mechanical properties of coupling agent and SD_MDF filled thermoplastic composites

ID	Flexural Properties		Tensile Properties			IS (J/m)
	FS (MPa)	FM (MPa)	TS (MPa)	TM (MPa)	EB (%)	
Control_HDPE	24.57[1]	1348.57	11.87	591.96	2.56	29.27
	0.88[2]	15.17	0.15	13.44	0.18	1.32
Control_PP	30.91	1427.49	13.63	710.43	2.81	19.17
	1.53	25.75	0.40	8.06	0.21	0.16
A1	24.75	1490.40	14.59	592.25	3.29	24.88
	6.78	261.55	0.98	48.54	0.31	2.31
B1	24.68	1692.57	12.82	598.38	2.71	21.68
	1.72	41.41	0.14	9.90	0.18	3.60
C1	24.87	1510.66	13.03	608.44	2.80	22.90
	1.48	25.44	0.34	8.07	0.23	2.51
A2	31.88	1649.10	14.48	729.15	2.93	15.78
	1.88	79.02	1.85	28.53	0.67	1.05
B2	32.01	1803.72	16.43	695	3.07	20.22
	0.50	80.61	0.95	22.60	0.09	0.56
C2	32.65	1791.18	16.16	605.43	3.39	17.76
	1.36	123.86	1.14	20.24	0.14	2.17

(1) Mean, (2) Standard deviation FS: Flexural Strength, FM: Flexural Modulus, TS: Tensile Strength, TM: Tensile Modulus, EB: Elongation at Break, IS: Impact Strength

As shown in Table 2, the use of coupling agent improves the bonding between lignocellulosic material and plastics, thus increasing the bending strength values of all composite types. The use of MAPE and MAPP provided a small increase of 1% to 6% compared to control samples for HDPE and PP. In many studies, it has been proven that the coupling agent act as a bridge between the lignocellulosic filler and the polymer matrix (Sanadi *et al.* 1997, Lu *et al.* 2000, San *et al.* 2008). It is also stated that maleic anhydride in MAPP and MAPE provide a polar interaction such as acid-base interaction and covalent bonding to hydroxyl groups on lignocellulosic fillers (Felix *et al.* 1993, Sanadi *et al.* 1995, San *et al.* 2008). The use of coupling agent improved in the flexural modulus values of the the thermoplastic composites (Table 2). The best results were obtained with the use of 3% coupling agent. With using 3% MAPE, a 26% increase was achieved in the samples with HDPE. The use of 3% MAPP resulted in an increase of 26% for PP based samples.

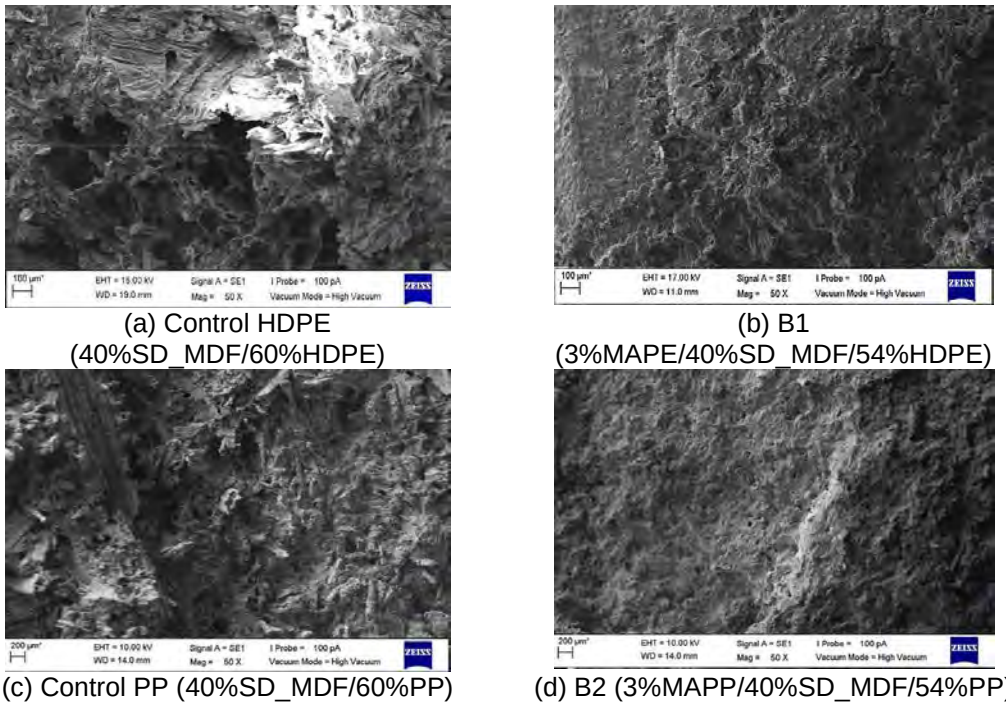
The tensile properties of the samples were positively affected when coupling agent was used in the polymer matrix. As the use of coupling agent improves the bond between lignocellulosic material and plastics, tensile strength values of all composite types have increased (Table 2). An increase of 23% compared to the control samples was determined with 3% MAPE in HDPE samples. In PP-produced samples, an increase of 21% was achieved with 3% MAPP use. Many studies have been

carried out on the investigation of the effects of the coupling agents on the mechanical properties of thermoplastic composites. Studies have shown that coupling agents increase the strength properties of composites (Felix *et al.* 1993, Sanadi *et al.* 1995, Sanadi *et al.* 1997, Lu *et al.* 2000, Li and Matuana 2003; Xu and Shuai 2007, Zhang *et al.* 2007, San *et al.* 2008, Mengeloglu and Karakus 2008). The use of MAPE and MAPP resulted in a 3% increase in tensile modulus values for two types of thermoplastics. Despite the fact that many studies have shown that there is a significant increase of 2-3 times in the properties of composites by increasing coupling agent use rates, there is no expected increase in the test results (Lu *et al.* 2000, Li and Matuana 2003, Xu and Shuai 2007, Zhang *et al.* 2007, San *et al.* 2008). However, in some studies, it has been reported that the significant improvement effect on the elasticity modulus properties was not observed when using coupling agent (Doan *et al.* 2006), and also indicated the values decrease in case of high usage of coupling agent (Santos *et al.* 2009). This situation is linked to two reasons. The first reason; the melt flow indexes of polyolefins grafted with maleic anhydrides are much higher than the melt flow indexes of polyolefins such as PP and PE. Therefore, it has a shorter polymer chain and lower molecular weight. Accordingly, use of coupling agent at high ratios may result in reduced modulus of elasticity. The second reason is that when MAPE and MAPP are used at high ratios, the thermoplastic polymer can be moved away from the lignocellulosic material and lead to weaker bonding and low resistance properties (Mohanty *et al.* 2006; Santos *et al.* 2009). The use of MAPE and MAPP also resulted in a small increase in the amount of elongation of the samples. Mengeloglu and Kabakci (2008) used 4% MAPE in eucalyptus wood flour filled thermoplastic composites and investigated the effects of coupling agent use on the properties of the composites. The results have indicated that MAPE use did not have a significant effect on the properties, causing a small increase in the amount of elongation at break.

In contrast to other mechanical properties, impact strength of the samples decreased with MAPE and MAPP use. Myers *et al.* (1993) have studied the effect of use of MAPP on the properties of PP-wood flour composite. They found that MAPP use had a negative impact on notched impact strength of the composites.

Morphological Properties

Fig. 3 shows SEM micrographs of SD_MDF and coupling agent filled thermoplastic composites at 50X magnifications. Some images show that the use of the coupling agent improves the bond between the lignocellulosic filler and the thermoplastic matrix. It was observed that the voids in the samples decreased and a more homogeneous distribution of the lignocellulosic material in the thermoplastic matrix was observed with using MAPE and MAPP by 3% compared to control samples.



(a) Control HDPE (40%SD_MDF/60%HDPE)

(b) B1 (3%MAPE/40%SD_MDF/54%HDPE)

(c) Control PP (40%SD_MDF/60%PP)

(d) B2 (3%MAPP/40%SD_MDF/54%PP)

Fig. 3.

Coupling agent at 3% concentration effect on morphological properties of thermoplastic composites filled with SD_MDF.

CONCLUSIONS

1. TS and WA values of the composites increased with increment of the water immersion time. With the use of coupling agent, it was seen that TS and WA of the composites decreased in comparison with the control samples. The best results for TS and WA were obtained on composites produced using 3% coupling agent.
2. The use of coupling agent improves the bond between the lignocellulosic material and the thermoplastic, resulting in an increase in flexural strength and flexural modulus values for all composite types. The best results were seen with use of coupling agent by 3%.
3. With the use of MAPE and MAPP, tensile properties including of the tensile strength, tensile modulus and the elongation at break of the composites were increased.
4. In contrast to other mechanical properties of the composites, there is generally a slight decrement in impact strength with MAPE and MAPP use.

ACKNOWLEDGEMENT

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DETERMINATION OF SELECTED PROPERTIES OF PP BASED COMPOSITES FILLED EGGPLANT (*Solanum melongena*) STALKS

İbrahim Halil BASBOGA
Kahramanmaras Sutcu Imam University
Faculty of Forestry, Department of Forest Industry Engineering
Address: KSU Avsar Yerleskesi. 46100 Kahramanmaras. Turkey
E-mail: ihbasboga@ksu.edu.tr

İlkay ATAR
Kahramanmaras Sutcu Imam University
Faculty of Forestry, Department of Forest Industry Engineering
Address: KSU Avsar Yerleskesi. 46100 Kahramanmaras. Turkey
E-mail: iatar@ksu.edu.tr

Kadir KARAKUS
Kahramanmaras Sutcu Imam University
Faculty of Forestry, Department of Forest Industry Engineering
Address: KSU Avsar Yerleskesi. 46100 Kahramanmaras. Turkey
E-mail: karakus@ksu.edu.tr

Fatih MENGELOGLU
Kahramanmaras Sutcu Imam University
[1]Graduate School of Natural and Applied Science, Materials Science and Engineering
[2]Faculty of Forestry, Department of Forest Industry Engineering
Address: KSU Avsar Yerleskesi. 46100 Kahramanmaras. Turkey
E-mail: fmengelo@ksu.edu.tr

Abstract

Composite materials are produced by combining two or more different components to form an individual product with better properties. Manufactured new composite materials may provide better performance than one component can provide by itself. Wood-plastic composites are manufactured by using thermoplastics and annual plant waste. In this study, utilization of eggplant (solanum melongena) stalks as a filler in manufacturing of injection molded PP based composites was investigated. For this purpose, neat polypropylene test samples (control group) and eggplant stalks flour-filled thermoplastic composites with different rates (7.5% 15% and 30%) were produced by using single-screw extruder and injection molding machine. Maleic anhydride polypropylene (MAPP) was utilized as coupling agents. Paraffin wax was used as a lubricant. Mechanical properties (tensile strength, flexural strength and impact strength) of produced composites were determined according to ASTM D 638, ASTM D 790 and ASTM D 256 standards. According to the statistical analysis, eggplant stalk flours had significant effect on tensile strength, tensile modulus, flexural strength, flexural modulus, elongation at break properties and density values. However, it had no significant effect on the impact strength properties. All produced composites provided standards requirements.

Key words: *injection molded; eggplant (solanum melongena) stalks; wood-plastic composite.*

INTRODUCTION

The usage of lignocellulosic fibers as fillers in the manufacturing of thermoplastic-based composites is increasing day by day. Moreover, the production of thermoplastic composites is a rapidly growing sector in the wood-plastic composite industry. A large variety of lignocellulosic wastes (wood flour, agricultural wastes etc.) and thermoplastic polymers such as polylactic acid (PLA), polyethylene (PE), and polypropylene (PP) can be used in the manufacture of composites. The reasons for preference of lignocellulosic fibers in thermoplastic composite are low cost, low density, no excessive wear during production, high specific strength, abundant and easy degradation in the environment. These advantages make it attractive for the usage of lignocellulosic materials in the production of thermoplastic composites (Bodirlu et al. 2009; Taj et al. 2007; Antich et al. 2006; Khalid et al. 2006, Georgopoulos et al. 2005; Renneckar 2004; Nair et al. 2001). In the previous studies, it has reported that various lignocellulosic fibers such as maple and spruce wood fibers, jute, hemp,

kenaf, sisal fibers, rice husks, wheat straw have been used in the production of thermoplastic composites (Poletto et al. 2011; Mengeloglu and Karakus 2008b; Mengeloğlu and Kabakcı 2008; Taj et al. 2007; Antich et al. 2006; Bengtsson and Oksman 2006; Digabel et al. 2004; Li and Matuana 2003). It has been determined that there is not enough study on the usage of eggplant stalks flour in the production of polypropylene based thermoplastic composite and there is a gap in that area.

With 18 million tons of eggplant production, China is the world leader. Turkey is in 4th place with 790 thousand tons of production. This amount accounts for 2% of world eggplant production. 30,000 hectares of eggplants are produced in Turkey (Topçu and Boyacı 2008). Eggplant is an annual plant species, dried plant stalks are collected and plant seedling is repeated in certain periods every year.

It is reported that 56 million tons of agricultural wastes are occurred in Turkey. (Karayılmazlar et al. 2011). These agricultural wastes are generally burned in the field and cause environmental pollution and reduce soil fertility (Çöpür, 2007). It is thought that these wastes can be utilized as a filler in the composite manufacturing. It is aimed to prevent environmental pollution and contribute to waste management.

OBJECTIVE

The main objective of this study was to determine how waste eggplant (solanum melongena) stalks have effect on mechanical properties of PP based composites. For this purpose, polypropylene based thermoplastic composites reinforced with waste eggplant stalk flour were produced. The mechanical properties of the manufactured composites were determined and results were compared with the ASTM D 6622 standard.

EXPERIMENTAL

In this study, polypropylene based thermoplastic composites reinforced with waste eggplant stalk flour were manufactured by injection molding method.

Material

Waste eggplant stalks were obtained from farmers in the Mediterranean region of Turkey. Waste eggplant stalk flours were used as lignocellulosic filler. Polypropylene (PP) which obtained from PETKİM was used as thermoplastic matrix. Maleic anhydrite grafted polypropylene ((MAPP) Licomont AR 504 by Clariant) were utilized as coupling agents. Descriptions of coupling agents were given in Table 1. Paraffin wax (K.130.1000) was used as a lubricant.

Table 1

Descriptions of the coupling agent used in this study

Descriptions	Licomont AR 504 (MAPP)
Appearance	Yellowish fine grain
Softening point	156°C
Acid value	41 mg KOH/g
Density at 23°C	0.91 g/cm^3
Viscosity at 140 °C	800 mPa.s

Method

Preparation of lignocellulosic filler material

Eggplant stalks obtained from the farmers in the Mediterranean region were granulated into flour form with Wiley Mill. Since the dimensions of the filler materials have important effects on the performance of the produced compsoites, the lignocellulosic flours screened and retained on 60 mesh-size screen (0.25mm) and 100 mesh-size screen (0.142mm), were used in this study. The classified fillers were dried in oven at 103 °C (±2) for 24 hours. It is important to use lignocellulosic fillers as a dried material in the manufacturing of polymer-based composites.

Manufacturing of polymer composites

The experimental design of the study was presented Table 2. Depending on the formulation PP, eggplant stalk flours (ESF), MAPP and paraffin wax were dry-mixed in a high-intensity mixer (900-1000 rounds per minute) to produce a homogeneous blend. These blends were compounded in a single-screw extruder at 40 rpm screw speed in the temperatures (barrel to die) of 170-175-180-185-190°C. Extruded samples were cooled in water pool and then granulated into pellets. The pellets were

dried in oven at 103°C (±2) for 24 hours (until moisture content reached approximately 0%). Dried pellets were injection moulded with 102 kg/cm² injection pressure and 80 mm/s injection speed to produce the test samples. The temperature used for injection moulded samples using an HDX-88 injection moulding machine was 180-200 °C from feed zone to die zone. After manufacturing, all tests samples were conditioned in a climatic room with the temperature of 20°C and the relative humidity of 65%.

Table 2

Manufacturing schedule of composites (%)

Group ID	Polypropylene (%)	Eggplant Stalk flours (ESF) (%)	MAPP (%)	Wax (%)
E1	100	0	0	0
E2	87.5	7.5	3	2
E3	80	15	3	2
E4	65	30	3	2

Testing of polymer composites

Testing of the samples was conducted in a climate-controlled testing laboratory. Densities were measured by a water displacement technique according to the ASTM D 792 standard. Tensile, flexural, impact strengths, were determined according to ASTM D 638 (5.0 mm/min), ASTM D 790 (2.0 mm/min) and ASTM D 256, respectively. Ten samples for each group were tested. Flexural and tensile testing were performed on Zwick 10KN while a HIT5,5P by Zwick™ was used for impact property testing on notched samples. The notches were added using a Polytest notching cutter by RayRan™.

Data analysis

The data of produced samples were analyzed by using Design Expert® Version 7.0.3. statistical program. ANOVA test was used to determine the effects of the factors.

RESULTS AND DISCUSSION

PP based eggplant stalk flour (ESF) filled composites were produced in the density range of 0.88-1.00 g/cm^3. The interaction graph of density was shown in Fig 1.

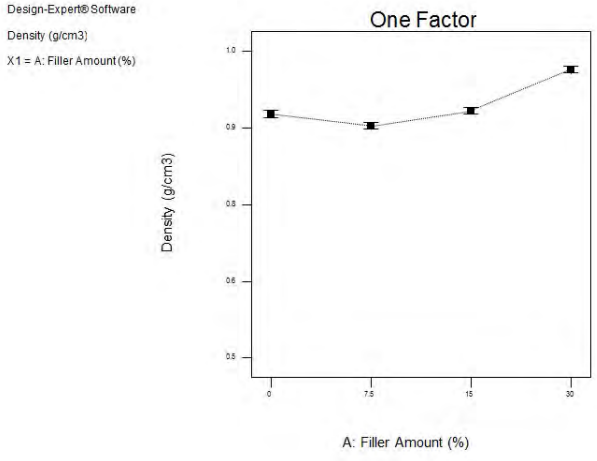


Fig. 1.
Interaction graphs of density.

When the interaction graph of density was examined, a slight increasing was observed in density values with the addition of ESF into the polymer matrix. However, this increasing wasn't statistically significant (P = 0.0918). In addition, the mechanical test results of the produced composite samples were summarized in Table 3.

Table 3

Mechanical properties of polymer composites

ID	Tensile Strength (MPa)	Tensile Modulus (MPa)	Elongation at Break (%)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Impact Strength (J/m)
E1	27.82 (0.42)*	495.24 (19.73)	460	42.36 (0.96)	1129.48 (25.56)	16.91 (2.73)
E2	25.13 (0.34)	568.04 (17.46)	14.3 (0.87)	45.17 (0.49)	1202.62 (49.90)	17.84 (3.14)
E3	24.90 (0.30)	669.87 (23.66)	9.35 (0.41)	47.31 (0.58)	1751.82 (23.43)	20.61 (4.54)
E4	24.33 (0.45)	792.82 (29.04)	5.21 (0.28)	48.16 (1.19)	2253.10 (89.11)	16.05 (2.88)

* Values in parenthesis are standard deviations.

The interaction graphs of tensile, flexural and impact strength test samples were given in Fig. 2-4, respectively. The tensile properties included tensile strength, tensile modulus and elongation at break. The interaction graphs of tensile strength, tensile modulus and elongation at break were given in Figures 2a, 2b and 2c, respectively.

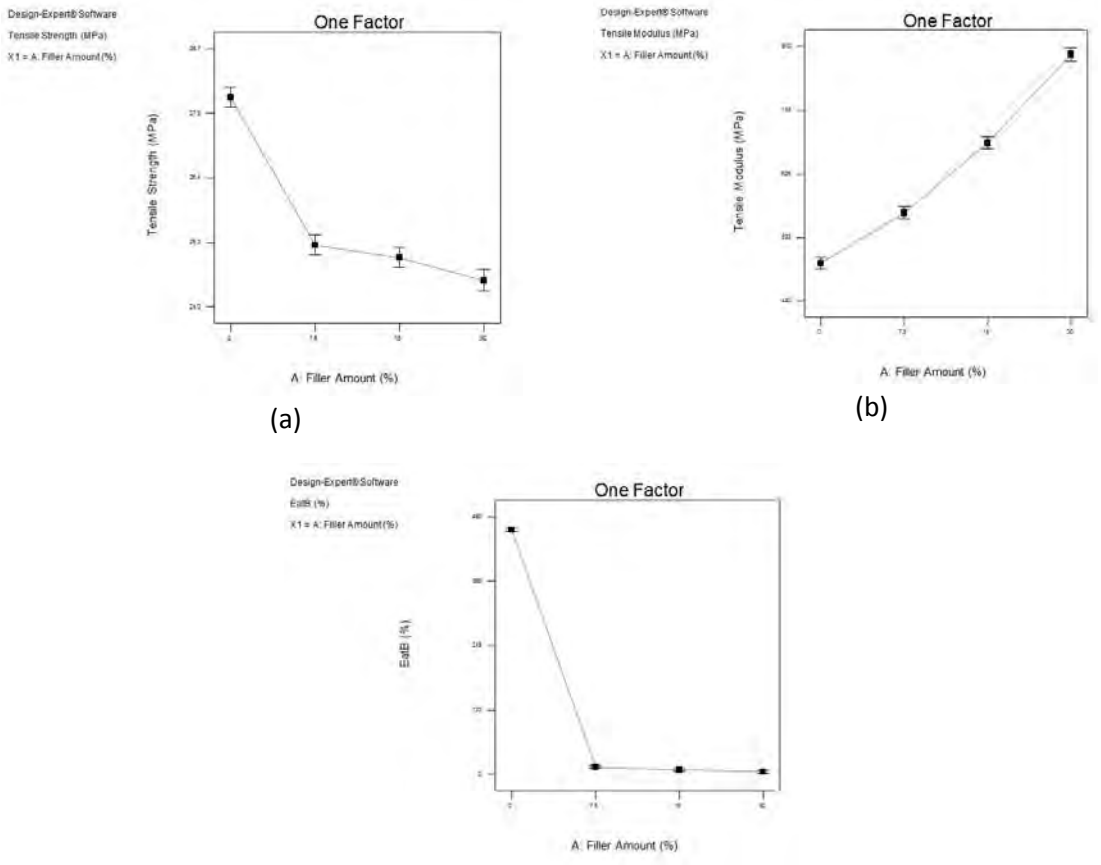


Fig. 2.
Interaction graphs of tensile properties
a) tensile strength, b) tensile modulus & c) elongation at break.

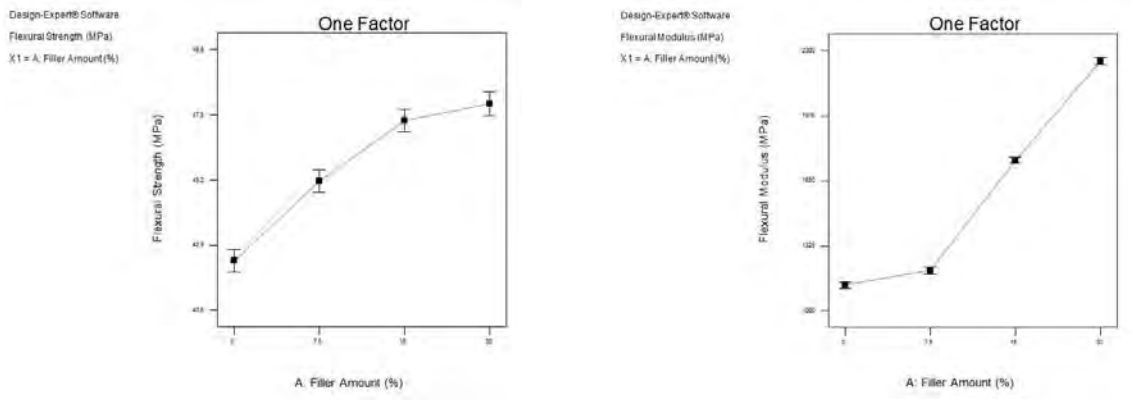
ANOVA (analysis of variance) were performed for tensile properties results of manufactured composite samples. According to ANOVA test, Eggplant stalk flours (ESF) rate had statistically significant effect on tensile strength ($P<0.0001$). Tensile strength was significantly reduced with

addition of ESF. It is thought that the most important reason of decline is lack of harmony between polymer matrix and lignocellulosic fillers (Balatinecz ve Woodhams 1993; Matuana ve Mengeloglu 2002).

To mention of tensile modulus, rise of ESF loading significantly increased the tensile modulus for composites (P<0.0001). Similar results for other wood flours filled polymer composites were also reported (Averous et al. 2000; Wang et al. 2003; Qiu et al. 2004; Mengeloglu et al. 2007). Lignocellulosic fillers have higher modulus than polymers. This is one of the advantages of the lignocellulosic fillers compare to polymer. That advantage leads to have higher modulus for wood plastic composites than unfilled polymer (Mengeloglu and Karakus 2008a). This can be explained by the rule of mixture (Matuana and Balatinecz 1998; Mengeloglu and Karakus 2008a).

ESF had statistically significant effect on elongation at break values (P<0.0001). With the addition of ESF, a decreasing in elongation at break was observed. Addition of the eggplant stalk flour into the PP polymer matrix, made the composites harder and more brittle. That was the reason why elongation at break values was reduced.

The flexural properties included flexural strength and flexural modulus. The interaction graphs of flexural strength and flexural modulus were shown in Figures 3a and 3b, respectively.



(a) (b)

Fig. 3.
Interaction graphs of flexural properties
a) flexural strength & b) flexural modulus.

Eggplant stalk flours (ESF) rate had statistically significant effect on flexural strength (P<0.0001). An increasing was determined in flexural strength with the loading of ESF. To mention on flexural modulus, a significant increase was observed like tensile modulus (P<0.0001). Eggplant stalks have a higher modulus values than plastic materials (Hornsby and ark. 1997). For this reason, when added to the polymer matrix, it increased the modulus values.

Results of the manufactured thermoplastic composites are compared with ASTM D 6662 (2001) standards. This standard is a standard for determining what the flexural strength values of polyolefin-based plastic lumber decking boards should be. Standard requirements are minimum 6.9 MPa for flexural strength and 340 MPa for flexural modulus. In this study, flexural strength values of the manufactured composites were found between 44.53-49.71 MPa. All the produced composites were reached standard requirements. To mention on flexural modulus values, the results of the entire composite produced with ESF were determined between 1123-2453 MPa. In parallel with flexural strength properties, all the produced composites were satisfied 340 MPa which requested from ASTM D 6662 (2001) standard.

The interaction graph of impact strength was given in Figures 4. According to these results, eggplant stalk flour ratio did not have statistically significant effect on impact strength (P=0.0918).

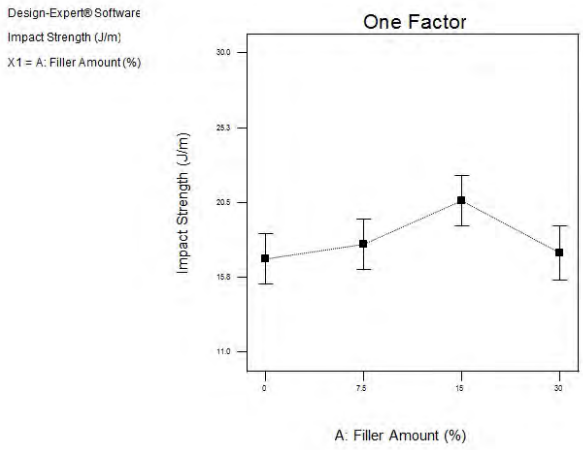


Fig. 4.
Interaction graphs of impact strength.

CONCLUSIONS

Polypropylene (PP) based polymer composites filled with different ratios of eggplant stalk flour were successfully manufactured by injection molding method. Density and mechanical results of manufactured composites were determined. According to results the following conclusions were reached;

- ✓ The eggplant stalk flour was significantly effective on the tensile strength and elongation at break properties of polypropylene based thermoplastic composites, with the increase of ESF rate in the composite, these properties were decreased,
- ✓ In contrast to tensile strength and elongation at break, with the addition of ESF, flexural strength, flexural modulus and tensile modulus values were improved. This increase was statistically significant.
- ✓ A decline was determined on the impact strength with the rise of ESF rate into the composites. However, this decrease was not statistically significant.
- ✓ All produced composites in this study provided flexural strength and flexural modulus values well over required ASTM D 6662 (2001) standards.

As a result, eggplant stalk flours were utilized as filler in the manufacturing of PP based composites. The utilization of these wastes in the production of thermoplastic composite materials might provide a new source of income for farmers. In addition, it might help to prevent from environmental pollution and reduce in soil fertility caused by burning of this kind of annual agricultural waste in the field.

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EFFECTS OF $CaCl_2$ AND NaCl ON STRENGTH AND SORPTION PROPERTIES OF CEMENT-BONDED RATTAN COMPOSITE PANELS

Abel O. OLORUNNISOLA
Department of Wood Products Engineering, University of Ibadan, Nigeria
Tel: 234 803 479 4945, E-mail: abelolorunnisola@yahoo.com

Subodh P. Agrawal
Retired Scientist, Organic Building Materials Division
Central Building Research Institute, Roorkee, India

Abstract

The aim of this study was to compare the effects of calcium chloride ($CaCl_2$) and sodium chloride (NaCl) as accelerators on the strength and sorption properties of cement-bonded composites produced from hammer-milled rattan cane (Laccosperma secundiflorum) fibre particles. 150 x 150 mm composite boards were manufactured at cement:rattan ratios of 4:1, 4.5:1 and 5:1 at a target density of 1000kg/m^3. Only bending strength, water absorption and thickness swelling properties were evaluated. Addition of $CaCl_2$ and NaCl improved board properties to varying degrees, depending on cement-rattan mixing ratio and salt concentration. Both accelerators enhanced the Modulus of rupture (MOR) of the composites particularly at 4% concentration. However, the MOR of the samples treated with 4% $CaCl_2$ were higher (4.4 – 6.91 MPa) than those of the samples treated with 4% NaCl (2.54 - 3.65 MPa). Water absorption by the untreated samples at 24 hours (36.7% - 46.%) was reduced by both $CaCl_2$ (24.1 – 36.3%) and NaCl (30.6 – 38.7%). The thickness swelling (TS) of all composites at 24 hours, except for samples produced with cement-rattan ratio of 4.5 to 1 and 2 % NaCl, fell within acceptable limit (< 2%). It was concluded that NaCl at 4% concentration is a possible substitute for $CaCl_2$ in cement-bonded rattan composite manufacture.

***Key words**: rattan cane; cement composite; calcium chloride; sodium chloride.*

INTRODUCTION

Cement-bonded composites (CBC) are low density products manufactured from a mixture of hydraulic cement and particles generated from lignocellulosics such as wood and agricultural residues. These products tend to combine the good qualities of cement, i.e., relatively high resistance to water, fire, fungus, and termite infestation; and good sound insulation with those of wood, i.e., high strength to weight ratio, nailability, and workability (Okino *et al*. 2005). They are used in building construction as fire resistant and acoustic panels (Wolfe and Gjinolli 1999, Ajayi 2006).

In recent years, the attention of researchers, particularly in the developing countries, has been focussed on the production of CBCs from agricultural fibres and residues (Savastano *et al.* 2001, Ajayi 2006, Roma Jr. *et al.* 2008). This is because large volumes of natural fibres generated in many of these countries, including Nigeria are largely under-utilised. Yet, many of them face the challenge of affordable housing provision for their teaming populations. Estimates show that housing deficit now stands at over 16 million units in Nigeria. An average of 1 million housing units per year is required not only to replenish decaying housing stock, but also to meet rising demand (Olorunnisola 2012).

Rattans are spiny, climbing, monocotyledonous palms generally found near water courses. Their distribution is largely confined to tropical and subtropical forests of Asia, Africa and the Pacific where they are usually collected almost exclusively from wild populations and used predominantly for furniture manufacture. The different parts of the plant are also used for basketry, mat making, binding, sporting goods (Ogunwusi 2012). However, a large volume of soft and immature portions of rattan cane is usually discarded during harvesting. Again, large quantities of fungus- infested/stained canes are usually discarded during furniture production. Cane wastage has been estimated by Liese (2002) at well over 30% of rattan harvested. Some of these considerable losses could be resolved with its alternative use for cement-bonded composite production as discussed by Olorunnisola (2005).

Like many other lignocellulosics, rattan cane tends to inhibit cement hydration when mixed with Portland cement for CBC manufacture. Several chemicals are used as accelerators to increase in the rate of setting in such composites. However, calcium ($CaCl_2$) is most widely used due to its predictable performance characteristics and successful application in concrete works over several decades. Addition of $CaCl_2$ improves cement curing by reducing moisture loss through evaporation during early hydration period by releasing the normal heat of hydration earlier and by accelerating the hydrating

action. Sodium chloride is an ionic compound made up of equal numbers of positively charged sodium and negatively charged chloride ions. Early studies by Henry and Griffin (1964) on sodium chloride in mixing water reported that it caused increase in the compressive strength of concrete at concentration of 25gm per 1kg of solution, with accompanying reduction in water vapour transmission. It was, however, also reported that NaCl had erratic effects in concrete, causing set acceleration in some cements and retarding effects in others (Mattus and Gilliam 1994). Shi *et al.* (2011) also reported that de-icers containing NaCl caused substantial compressive strength loss in concrete. However, no study has been reported so far on the effect of NaCl, a readily available and relatively cheap salt, on the bending strength and sorption properties of CBCs, though both salts are well known for their water retention capacity which tends to impact positively on cement curing.

OBJECTIVE

The main objective of this study, therefore, was to compare the effects of the addition of $CaCl_2$ and NaCl at different levels of concentration on the bending strength, water absorption and dimensional stability of rattan cement-bonded rattan composites.

METHOD, MATERIALS AND EQUIPMENT

Freshly harvested, rattan cane (*Laccosperma secundiflorum*) samples obtained from rattan processors were air-dried for three weeks to reduce their moisture and sugar contents, hammer-milled into fibre particles and and used in composite production 'as received'. The properties of the $CaCl_2$ and NaCl as provided by their manufacturers are presented in Table 1.

Table 1

The Chemical Compositions of the $CaCl_2$ and NaCl Salts

Salt	Property	Value
$CaCl_2$		
	Assay	90% min.
	Iron	0.002% max.
	Sulphate	0.05% max.
	Heavy mteals (e.g. lead)	0.002% max.
	$CaCl_2$ (molecular weight)	110.99
NaCl		
	Sodium	38.7g/100g
	Iodine	>15 ppm

The oven-dry moisture content of the "as received" rattan cane fibre particles was determined in accordance with BS 812-109 (1990), while sieve analysis was carried out with a set comprising 2.36 mm, 1.7mm, 1.18mm, 0.85mm, 0.6mm and 0.045mm sieves in accordance with BS 812-103 (1990). To determine water absorption by the 'as received' rattan cane fibre particles, 20g of the fibres were completely immersed in 300 ml of distilled water. The soaked fibre particles were filtered after 24 h and washed with distilled water. The fibre particles were weighed after draining off the excess water and the water absorption value in percentage was then computed as in Aggarwal *et al*. (2008). Two replicates were used and the mean values of the results obtained are reported.

For composite manufacture using $CaCl_2$ and NaCl as chemical accelerators for the different sets of triplicate samples, each salt was dissolved in the water used for the mixing process in two proportions by mass of cement, i.e., 2 and 4 %. For the control samples, no salt was added to the mixing water. The composite samples were then manufactured by manual dry-mixing of rattan particles and Type 1, general purpose Portland cement (class strength 42.5 grade) in a plastic container at three cement: wood ratios by mass, i.e., 4:1, 4.5:1 and 5:1 respectively. The potable mixing water was then added to the dry mixture based on the equation developed during preliminary studies on water requirements for rattan-cement composite mixtures:

$$Q = 0.36C \quad (1)$$

where: Q = Quantity of water (Millilitres)
C = quantity of cement in the mixture (grams)

Each wet mixture was poured into single units of 150 x 150 x 25 mm metallic moulds, placed in a hydraulic cold press set at a pressure of 6.6 N/mm^2 and pressed for 6 to 8 hours, but left in the

mould for 24 hours. Once de-moulded, the composites were cured at ambient room temperature (20±2^{0}C) under wet towels for the first seven days, and then in a chamber maintained at a constant temperature and relative humidity of 25±2^{0}C and 65±5% respectively for 21 days. Three specimens from each mixture were tested at 28 days. The oven dry moisture content, density and bending Modulus of Rupture (MOR) of the samples were determined in accordance with Indian standard, IS 14862 (2000). The 3-point bending test was conducted on a 20 kN capacity Universal Testing Machine (Shimadzu, Model AGS2000G) adopting a span of 100 mm and mid-span deflection rate of 1.0 mm/min.

To determine water absorption (WA) in, and thickness swelling (TS) of the rattan-cement composites, three 150x150mm specimens each were thoroughly sand-papered and dried in an electric oven set at 60±5^{0}C until constant weight (≤0.1% weight change) was achieved. The specimens were then brought to room temperature (25±2^{0}C) at a relative humidity of 65±5%. This drying method was selected to minimize any modification to the capillary pore structure that may be caused by a higher temperature and more rapid drying (Guneyisi and Gesoglu 2008). The dry mass and thickness of each specimen were first measured and recorded. The specimens were then completely immersed horizontally in potable water maintained at a temperature of 20±2^{0}C. Water Absorption after 1 and 24 hours respectively were calculated from the increase in weight of the specimen during submersion, while the Thickness Swelling of each board was expressed as a percentage of the original thickness. All composite property test results were subjected to analysis of variance procedure for 2-factorial experiment at 5% level of significance.

RESULTS AND DISCUSSION

Physical Characteristics of the Wood Particles

Table 2 shows the 'as received rattan cane fibre particle distribution. Close to 80% of the fibre particles were retained on sieve sizes ranging from 0.6 and 0.045 mm, an indication that the rattan fibres were relatively small. The mean water absorption at 24 hours was 365.8%. This value compares favourably with those reported by Agopyan (1988), Aggarwal *et al.* (2008), and Olorunnisola and Agrawal (2009) for different types of wood and vegetable fibres used in cement composites, i.e., *Eucalyptus Tereticornis* (286 - 433.0%), sisal fibre (239%), jute (214 %), piassava fibre (34.4 – 108%), coir fibre (117-171%), banana (400%), aak (350%), castor (235%), bhabar (185%), bamboo sticks (145%), arhar flakes (170 -200%), and arhar powder (250-320%) It can be inferred, therefore, that the rattan cane fibre particles were highly hygroscopic.

Table 2

Sieve Analysis of the 'As Received' L. secundiflorum Fibre Particles

Sieve Aperture (mm)	Retained Particles (%)	Cumulative Particles retained (%)
2.36	0.72	0.72
1.70	5.42	6.14
1.18	0.84	6.98
0.85	13.32	20.30
0.60	24.17	44.47
0.045	53.72	98.19
Pan	1.82	100.0

Density of Cement-Bonded Rattan Composites

The densities and the oven dry moisture contents of the composite samples are shown in Fig. 1 and Fig. 2 respectively. The density values ranged between 1.06 and 1. 28g/cm^3, i.e., 1060-1280Kg/m^3 while the moisture content ranged between 4.0 and 11.6%. The density of all the composite samples exceeded the minimum value of 1000Kg/m^3 stipulated in ISO 8335 (1987) for cement-bonded composites. Expectedly, there was a general increase in density with increase in cement content. While the addition of $CaCl_2$ contribued to a slight increaase in the density of the composite samples, while the addition of NaCl did not. The moisture contents of the samples were below the 12% maximum value specified for cement-bonded composites in ISO 8335 (1987). Analysis of variance (ANOVA) presented in Table 3 also showed that the addition of both $CaCl_2$ and NaCl as chemical accelarators resulted in a signficant incease in the moisture content of the composite samples. This was expectedly due to the well-known water retention property of both salts. The positive implication of the water retention is that cement hydration would be enhanced.

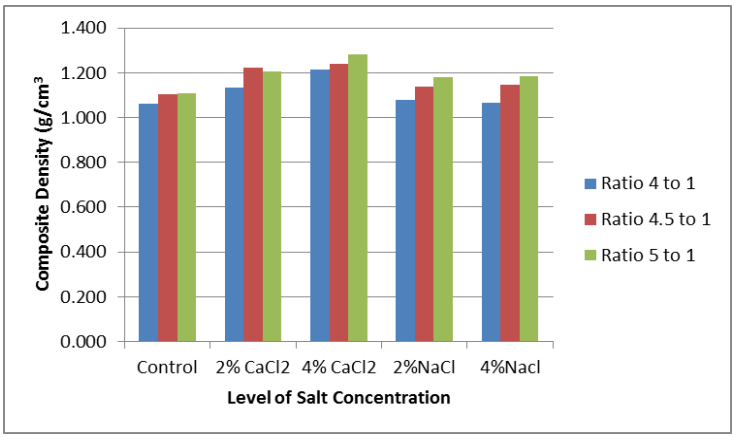

Fig. 1.
The densities of the composite samples.

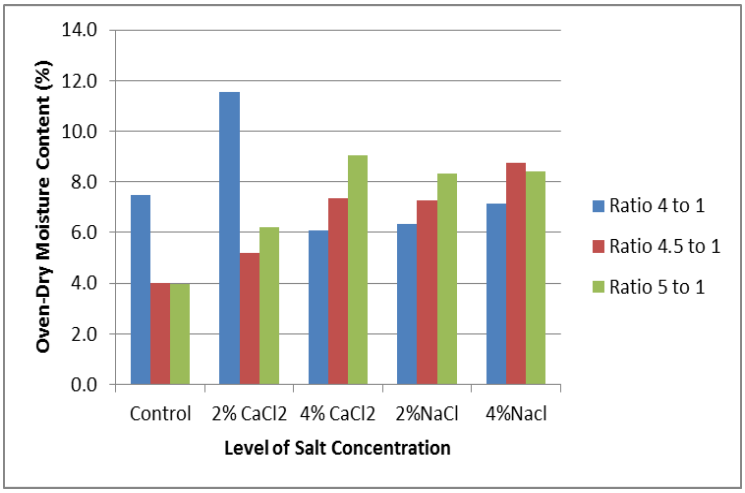

Fig. 2.
The oven dry moisture contents of the composite samples

Table 3
ANOVA on Effects of Cement-Rattan Ratio and Accelerator on Composite Moisture Content

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Cement : Rattan Ratio	10.864	2	5.432	8.102	0.001537	3.315
Accelerator	48.221	4	12.055	17.980	1.25E-07	2.689
Interaction	107.514	8	13.439	20.045	4.64E-10	2.266
Within	20.113	30	0.670			
Total	186.712	44				

Modulus of Rupture

As shown in Fig. 3, the MOR of the composites ranged between 0.86 and 6.91MPa. These values generally compare favourably with the range of values (1.7 to 5.5MPa) reported by the Forest Products Laboratory (1999) for several kinds of low density (500 to 1000kg/m^3) cement-bonded particleboards. The MOR values of the samples treated with 4% $CaCl_2$ were generally higher (4.4 – 6.91MPa) and compare favourably with the range of values (5.8 to 6.4MPa) reported by Okino, *et al.* (2004) for cement-bonded particleboard produced from a mixture of eucalyptus and rubberwood. However, all the MOR values recorded fell below the minimum of 9.0MPa stipulated in ISO 8335 (1987) for cement-bonded composites. The cement-rattan mixing ratio had no significant effect on the MOR as shown in the ANOVA in Table 4.

Modulus of rupture is an index of the maximum load-carrying capacity in bending when cement-bonded composite products are used as panels in ceiling and roofing where they are subjected to flexural stresses. It's value depends primarily on the bonding strength between the aggregate material and cement. The relatively low values obtained in the present study suggest that the cement-bonded rattan composites could be used for ceiling and such allied applications.

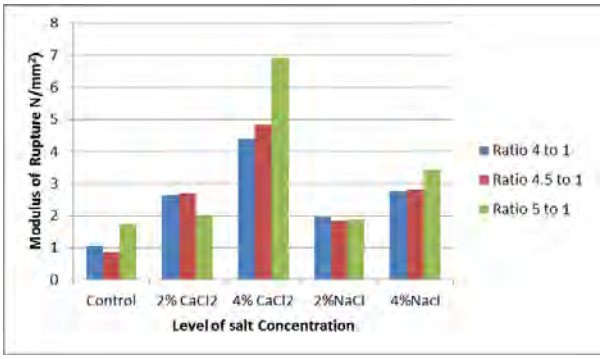

Fig. 3
The Modulus of Rupture of the composite samples.

Table 4

ANOVA on Effects of Cement-Rattan Ratio and Accelerator on MOR

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Cement: Rattan Ratio	3.76572	2	1.88286	4.621171	0.017803	3.31583
Accelerator	91.40237	4	22.85059	56.08302	1.69E-13	2.689628
Interaction	10.02712	8	1.253391	3.076241	0.011818	2.266163
Within	12.22327	30	0.407442			
Total	117.4185	44				

Water Absorption

The test results for WA at 1 hour and 24 hours respectively are presented in Fig. 4 and Fig. 5. The WA values ranged between 15.0 and 38.8 % and between 24.1 and 46.1 % after 1h and 24h of immersion respectively. It is clear from the 1 H test results that the cement-bonded rattan composite samples had a relatively high water absorption capacity, and may therefore not be installed outdoors. The control (untreated) samplesexhibited the highest WA, regardless of the rattan-cement ratio, perhaps due to their relatively low moisture content and the presence of more void spaces within the composite. The $Cacl_2$-treated samples generally absorbed less water than the and NaCl-treated samples, particularly at 4% level of concentration.

Thickness Swelling

The 1h and 24h TS values obtained for the composite samples ranged between 0.2 and 1.7 %, and between 0.4 and 2.1 % respectively (Fig. 6 and Fig. 7). Except for the samples produced using

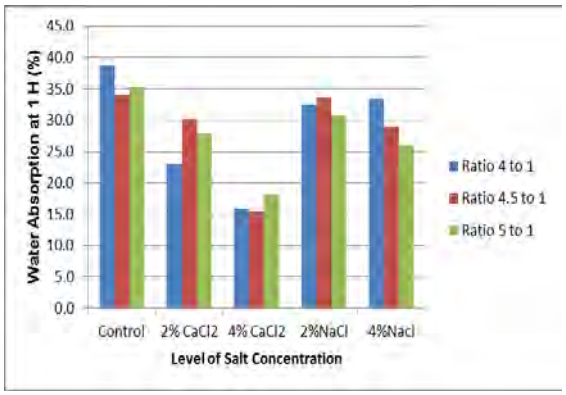

Fig. 4.
Water absorption by the composite samples at 1 Hour.

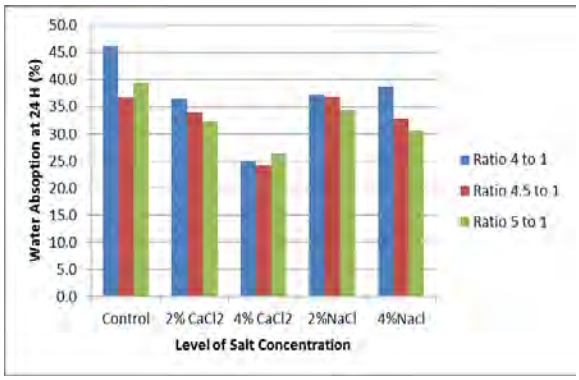

Fig. 5.
Water absorption by the composite samples at 24 Hour.

the cement-rattan mixing ratio of 4.5 to 1 and 2 % NaCl as chemical accelerator, the TS values at 24h were quite low and satisfied the maximum TS values of 1.8 % and 2.0 % specified by BS 5669 (1989) and ISO 8335 (1987) standards respectively for cement-bonded composites. The addition of $CaCl_2$ at 4% level of concentration had the greatest effect on the the reduction of TS, while NaCl addition resulted in a genral increase in the TS. However, since the increses observed were still within acceptable limits, bith As shown in the ANOVA presented in Table 6, wood-cement ratio and the combined effects of wood-cement ratio and pre-treatments had significant effects on 24- TS.

CONCLUSIONS

Wood-Cement composites were produced from rattan (*Laccosperma secundiflorum*) fibrous particles. The composites were tested for bending strength, water absorption and thickness swelling. Results obtained showed that calcium chloride had a more positve effect than sodium chloride on the flexural strength, water absorption and thickness swelling of cement-bonded rattan composites. However, given its acceptable performance, the use of sodium chloride is recommended as a substitute for composite manufacture if calcium chloride is not available. .

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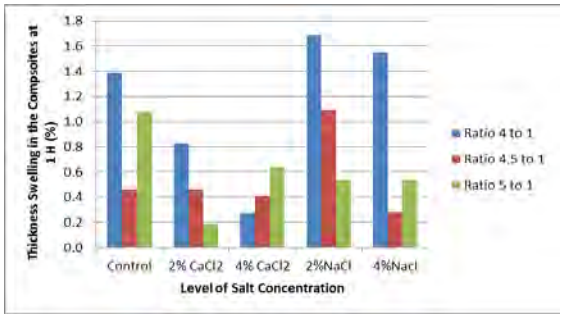

Fig. 6.
Thickness swelling of the composite samples at 1 Hour

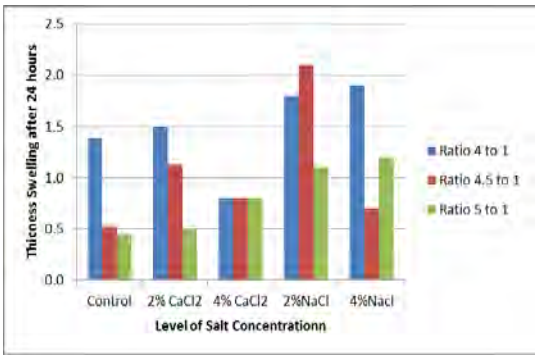

Fig. 7.
Thickness swelling of the composite samples at 24 Hour

Table 6

ANOVA on Effects of Cement-Rattan Ratio and Accelerator on 24 H WA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Sample	155.9743	2	77.98714	6.339014	0.005055	3.31583
Columns	1152.719	4	288.1798	23.42406	7.3E-09	2.689628
Interaction	143.0197	8	17.87746	1.45313	0.215911	2.266163
Within	369.0817	30	12.30272			
Total	1820.795	44				

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RESEARCH INTO THE PROPERTIES OF AN INNOVATIVE WOOD-PLASTIC COMPOSITE

Cosmin SPÎRCHEZ
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 415315, Fax: 0040 268 415315, E-mail: cosmin.spirchez@unitbv.ro

Aurel LUNGULEASA
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: lunga@unitbv.ro

Abstract

The paper to analyses the characteristics of an innovative type of wood-plastic composite with a low wood content. Using traditional methodologies based on standardized European test methods, the density, strength and modulus of elasticity in static bending were determined. Additionally the impact strength of the composite was analysed by employing the method with two pendulum hammers. Good results were found for their physical and mechanical properties. In conclusion the paper suggests that the analysed product had good properties for specific uses in environments with high humidity.

Key words: *density; impact; modulus of resistance (MOR); wood-plastic composites (WPCs).*

INTRODUCTION

Wood-plastic composites (WPCs) are new composites with multiple uses, utilized especially in high humidity environments or with direct water contact. Wood-plastic composites are nano-composites of the last generation, usually made of a wooden matrix to which plastic is added as a reinforcing filler. The reverse combination wood-plastics is also possible. Usually, WPC_s have a ratio 1:1 wood/plastic and almost insensible to humidity/water. Manufacturers guarantee strongly for these products, affirming that wood-plastic composites are more environmentally friendly and require less maintenance than the alternative use of solid wood treated with preservatives or some rot-resistant species. Moreover, WPC_s have very good physical and mechanical properties, and their price is acceptable, as is visible in Table 1.

Table 1

Physical and mechanical properties for wood plastic composites using wood in combination with glass fibres and thermoplastic resin (Barbu, 1999)

Property	Product A	Product B	Product C
Density, kg/m^3	1300	1500	1400
Wood content, %	60	35	-
Fibre glass content, %	-	35	50
Thermoplastic resin content, %	28	23	40
Modulus of resistance (MOR) to bending, MPa	69	103,5	117,3
Modulus of elasticity (MOE) to bending, GPa	1,32	7,9	11,0
Resistance to traction parallel to planes, MPa	69	75,9	69
Expansion after 2 hours, %	7,7	1,3	6,6

WPCs are obtained from virgin or recycled plastic mixed with wood or other natural fibres. In the last 10-15 years, these composites have experienced a great development in terms of research, production and distribution, but mostly in understanding how to obtain certain materials with the desired characteristics. The most common uses of these composites are found in outdoor floorings, but they can also be used for transverse beams, gates, fences, garden and park benches, door and window frames and indoor furniture, mostly in pools, Olympic-size swimming pools, bathrooms and kitchens (Fig.1).

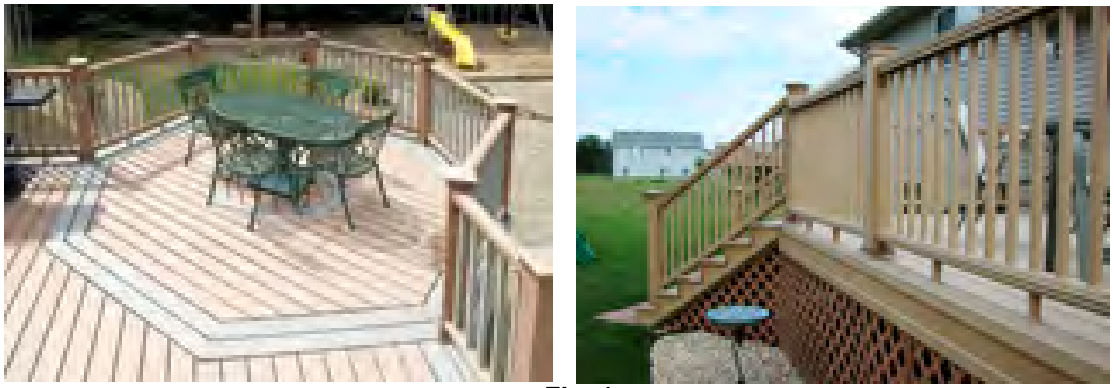

Fig. 1.
Some uses of the wood-plastic composite (www.wpcromania.ro).

At this time, the automotive industry is the largest user of WPCs in Europe with over half of the overall usage. It is also estimated that many European automotive companies will diversify the wood-plastic composites, using flax and hemp natural fibres, which are longer and more resistant than wood fibres. On the other hand, there is another purpose, namely of introducing the composites to the construction and furniture markets.

MATERIALS AND METHODS

The analyzed material is an innovative wood-plastic composite made of 80% plastic, 10% powdered chalk and colorants and 10% woody sawdust. This composite was purchased as panels with an area of 600 x 600mm and thickness of 12, 15, 18 and 21mm from the manufacturing company Kompozite Holz from Brasov, Romania. These panels have been cut into 20 replicates for each thickness in order to determine the density (50 x 50mm), for static bending 20 samples of 290 x 50mm, 350 x 50mm, 410 x 50mm and 470 x 50mm, depending on the thickness of the panels, 10 samples each for water absorption and swelling in thickness and 10 samples of 150 x 150mm for determination of impact strength.

In order to determine the density of the samples the classical method was used for reporting the mass of the sample relative to its volume (according to the European standard EN 323:1993).

$$\rho = \frac{m}{l \cdot w \cdot t} \left[\frac{kg}{m^3}\right]$$

where:
m is sample mass, expressed in g;
l - Length of sample, in mm;
w - Width of sample, in mm;
t - Thickness of sample, in mm.

The mass of the samples were determined with an electronic balance, expressed in grams accurate to 3 decimals. The three dimensions of the samples were determined with an electronic calliper accurate to 2 decimals. After calculations were made to determine the density expressed in g/mm^3, the result was converted into the international unit for density, namely kg/m^3. Next, using Excel Microsoft program, the two statistic parameters, arithmetic mean and square deviation, were determined.

Modulus of resistance (MOR) and modulus of elasticity (MOE) in static bending were determined using the methodology stipulated in the SR EN 310:1996. A universal testing machine was used with specific devices for such tests, namely two adjustable bearings on the bottom side to support the sample and a punch to apply force in the case of resistance and two punches for determining the modulus of elasticity in static bending. The distance between bearings was 20 times longer than thickness, as prescribed, and the speed with which the force was applied was 10mm/min.

The equation for determining the resistance was the following:

$$MOR = \frac{3}{2} \cdot \frac{F \cdot l}{w \cdot t^2} \left[N/mm^2\right]$$

where:
F is ultimate strength, expressed in N;
l - Distance between bearings, in mm;
w - Width of sample, in mm;
t - Thickness of sample, in mm.

The modulus of elasticity (MOE) in static bending is determined using the following equation:

$$MOE = \frac{l^3 \cdot \Delta F}{4 \cdot w \cdot t^3 \cdot \Delta f} [\mathrm{N/mm^2}]$$

where:
ΔF is difference in forces P_2-P_1, the first force being approximately 10% of the ultimate fracture force, and the second one being approximately 40% of breakage load, expressed in N;
Δf is the difference in deformation for the sample, f_2-$f_1$corresponding to the two above forces, expressed in mm.

20 samples were used to determine the resistance and the modulus of elasticity in static bending.

Impact strength was determined by several methods, namely the method with one strike using the Charpy pendulum impact (usually for solid wood), the method with repeated hammer strike (usually for panels) and the method with double pendulums (usually for panels). The working principle of the Charpy pendulum impact machine is shown in Fig. 2, where it can be seen how the pendulum is dropped from a known height from the starting position, hits and breaks the sample found at the bottom, then rises to a known height. This height, along with the initial height, is determined by the angle seen on the angle dial.

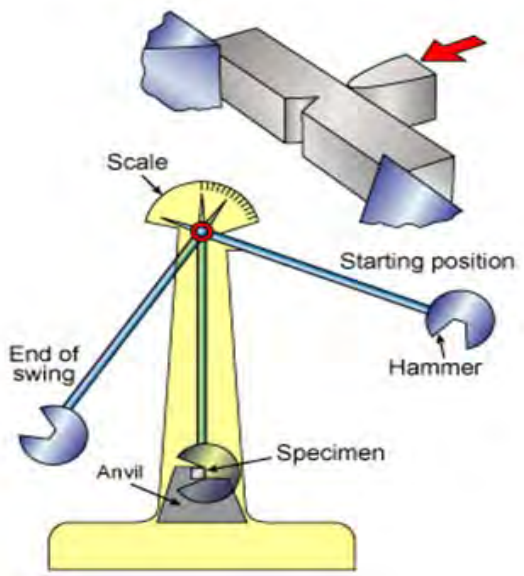

Fig. 2.
Charpy pendulum impact machine (IT 2017).

The double pendulum method is accurate and is specific for wooden and plastic panels, as well as for other composites. This method for determining the impact strength uses two pendulum hammers, but only one kicks to break the sample. The sample is square shaped, measuring 150mm. The machine is mainly composed of two pendulums, each with own dial and dial pointer, a blocking system for the active pendulum hammer, a power brake to stop the movement of the pendulums and the resistance frame (Fig. 3).

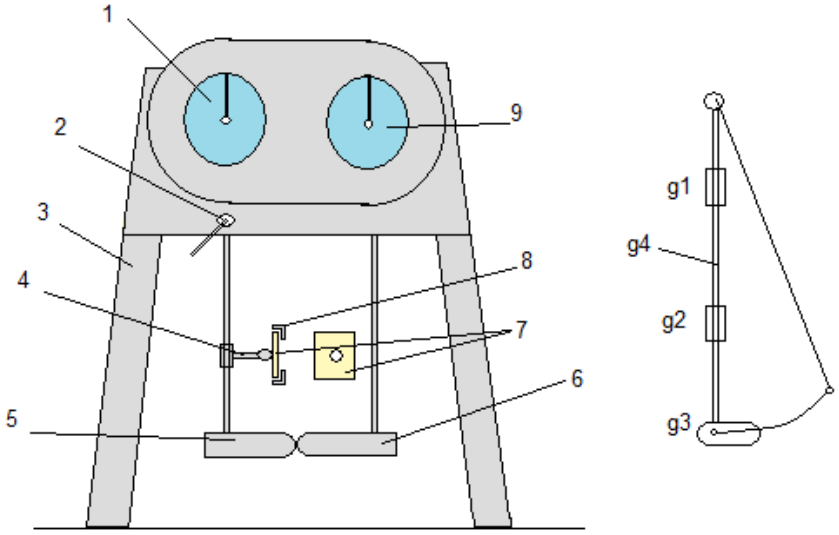

Fig. 3.
Double pendulum impact.

where:
1 is dial of active pendulum hammer;
2 - Handle for blocking the active pendulum hammer;
3 - Resistance frame;
4 - Blowing punch;
5 - Active pendulum hammer;
6 - Passive pendulum hammer;
7 -Wood-plastic composite sample;
8 - Sample frame;
9 - Dial of passive pendulum hammer;
g1- Mass of first weight, expressed in g;
g2- Mass of second weight;
g3- Mass of pendulum;
g4- Weight of arm.

The punch (4) has an oval shape so it not to rub against the sample and increase impact strength without reason. The active pendulum hammer (5), through its mass (g_3), as well as the other masses (g_2, g_1 and g_4), ensures the energy necessary to hit the sample, as well as fine adjustment. After the punch breaks the sample, the remaining energy of the active hammer is transferred by impact to the passive hammer which acquires an angular movement, shown on its dial.

The mechanical work performed by the active pendulum hammer depends on the mass, distance from the main pivot to the centre of gravity and on the angle from which the movement is initiated, the equation being the following:

$$L1 = G \cdot l_c \cdot (1-\cos \alpha) \text{ [daN·m]}$$

where:
G is the sum of the four weights' masses, approximately 17,8daN (hammer 8.2kg; additional weight 7.2kg; axle weight 1.9kg and punch weight 0.5kg, α – the angle from which the active pendulum hammer is launched;
l_c is distance from the main pivot to the centre of gravity of the entire unit (Mitisor 1977), calculated with the following equation (distance from the main pivot 800mm for hammer, 420mm for a additional weight and 600mm for punch):

$$l_c = \frac{\sum_{i=1}^{4} g_i \cdot l_i}{G}$$

For the mechanical work performed by the active hammer to 10daN·m, the angle is 90^0 the distance l_1 should be 8.57cm. The mechanical work spent for breaking the sample is determined as the difference among other mechanical works, respectively:

$$L=L_1-(L_2+L_3)$$

where:
L_1 is the mechanical work performed by the active hammer;
L_2 - the mechanical work of the passive hammer;
L_3 - the mechanical work spent through friction and transfer from one hammer to the other.

The mechanical work spent through friction and collision can be determined by performing a test without a sample, when $L_3=L_1-L_2$. It is recommended that the absorbed energy by piercing the sample should be around 25% of the active hammer's energy. Impact strength (resilience index) is determined as the ratio between the mechanical work spent for breaking the sample and the thickness of the sample, respectively:

$$I = \frac{L_1 - (L_2 + L_3)}{g} \qquad [daN \cdot m / cm]$$

A board's impact strength is determined as the arithmetic mean for the 10 pieces, cut from each type of board (each thickness separately).

In order to observe the influence of the water on this wood-plastic composite, some samples were cut into pieces of 50x50mm which were submerged in water for a period of 2 hours and absorption and swelling in thickness of samples were determined by employing standardized European methods (SR EN 317:1996).

RESULTS AND DISCUSSION

When analyzing the exterior appearance of the product, smooth, glossy the surfaces can be observed. The cuts performed with the circular blade are clear, even if the appreciable pore spaces can be observed to increase from the sides towards the panel's inner part. The panels are light and the density reached a mean value of 612.4kg/m^3, with a mean square deviation of 6.6kg/m^3 for the 12 mm thickness panel. This value is very low compared to other similar products (Barbu 1999) with densities over 1300kg/m^3. From this point of view, the analysed WPC_s product belongs to the light panel category. The influence of the thickness on the panels' density represents a slight increase, as shown in Fig. 4.

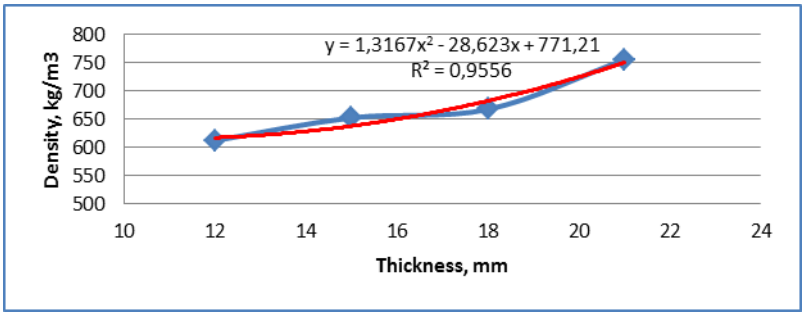

Fig. 4.
The influence of panel thickness on its density.

Modulus of resistance for bending strength obtained from laboratory tests was low, respectively 25.8N/mm^2 (with a standard deviation of 1.5N/mm^2) for a 21mm thickness of WPC_s, compared to 70-100N/mm^2 found by other authors (Fang et al. 2013). The low value of the static bending strength results from the lack of some stiffening agents in the structure of the panels, such as fibre glass.

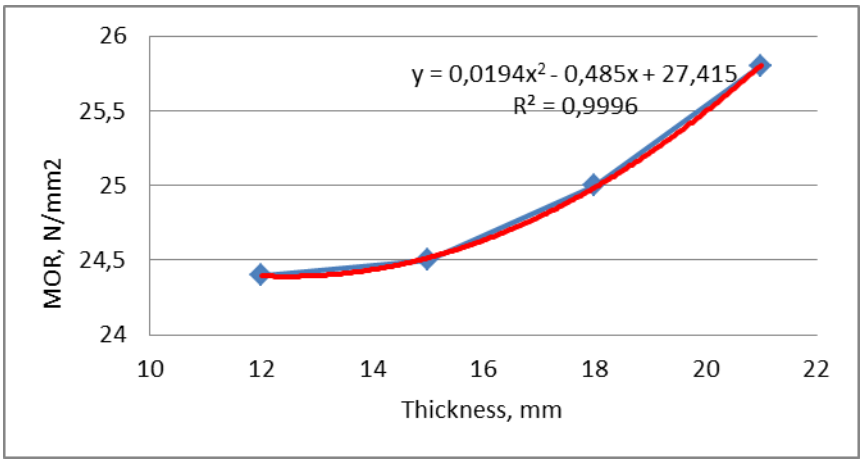

Fig. 5.
Slight influence of thickness on static bending strength.

The influence of thickness on static bending strength was slight (Fig. 5), respectively it increased along with the increase in panel thickness. This increase is due to the increase of panels' density given by the panels' thickness. Modulus of elasticity (MOE) in static bending had low values of 1540-1760N/mm^2, with a slight decrease depending on the panel's thickness (from 1766.5N/mm^2 for 12mm thickness, to 1543.3N/mm^2 for 21mm thickness of panel).

Referring to water absorption and swelling in thickness, there were no significant changes of size and mass of samples. Both water absorption and swelling in thickness having insignificant values of under 0.2%.

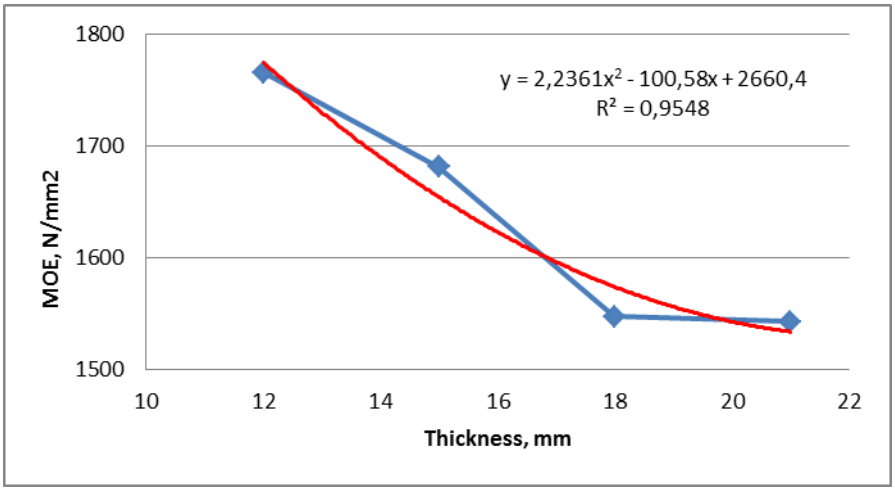

Fig. 6.
Modulus of elasticity related to panel thickness.

Impact strength or the index of resilience of composite panels depends on the mechanical work of the active and passive hammer, on the mechanic work lost through collision and friction and of the thickness of the panel. The lost mechanical work was constantly 4.031daN·m. The index of resilience of the analyzed composite is dependent on the thickness of the panel, having a significant increase from 2.3 to 6.1daN·m/cm (Fig. 7).

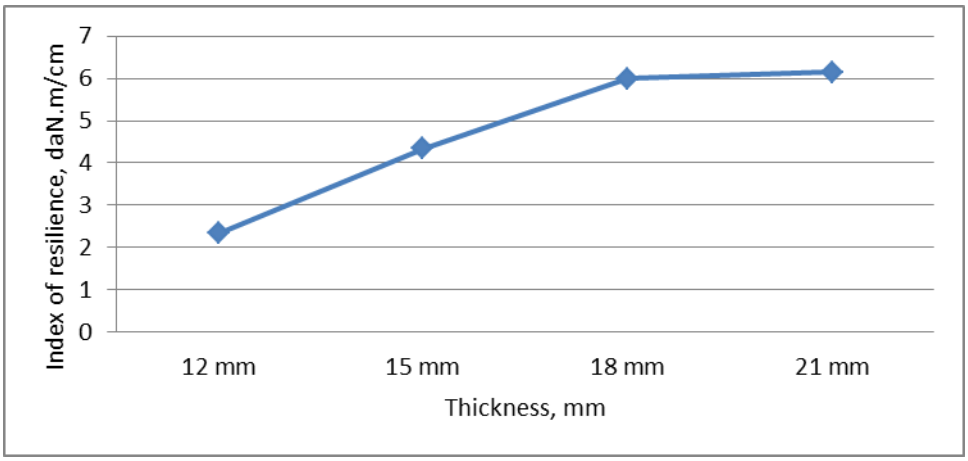

Fig.7.
Index of resilience related to panel thickness.

In order to make a comparison about index of resilience, there were conducted tests on 10 samples of structured chipboards with thickness of 15mm. The work consumed for breakage was 0.94daN·m and an index of resilience of 0.62daN·m/cm. The values for chipboard comparison are much smaller than of the wood-plastic composite that was analysed in the paper.

CONCLUSIONS

Wood-plastic composite panels with a low content of woody sawdust that have been analyzed in the present paper are porous panels with smooth and glossy surfaces. These composites have proven good resistances and potential to various uses. Impact strength values are very good compared to those of particle boards. Water behaviour is very good, in this case, the panels having the great properties of plastic. In what concerns the area of use, these wood-plastic composite panels can be successfully used in humid environments or in direct contact with water, especially since processing cuts are fine, without pulling out fibres or chips.

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THE EFFECT OF DIFFERENT TEST METHODS ON DURABILITY CLASSIFICATION OF MODIFIED WOOD

Gry ALFREDSEN
Norwegian Institute of Bioeconomy Research, Department of Wood Technology
P O Box 115, NO-1431, Ås, Norway
Tel: 0047 91876568, E-mail: gry.alfredsen@nibio.no

Christian BRISCHKE
University of Goettingen, Faculty of Forest Sciences and Forest Ecology,
Department of Wood Biology & Wood Products
Büsgenweg 4, D-37077 Göttingen, Germany
E-mail: christian.brischke@uni-goettingen.de

Linda MEYER-VELTRUP
Leibniz University Hannover, Faculty of Architecture and Landscape Sciences,
Institute of Vocational Sciences in the Building Trade
Herrenhäuser Str. 8, D-30419 Hannover, Germany
E-mail: meyer@ibw.uni-hannover.de

Miha HUMAR
University of Ljubljana, Biotechnical Faculty, Dept. of Wood Science and Technology
Jamnikarjeva 101, SL-1000 Ljubljana, Slovenia
E-mail: Miha.Humar@bf.uni-lj.si

Per Otto FLÆTE
Norwegian Institute of Wood Technology, P O Box 113 Blindern, NO-0314 Oslo, Norway
E-mail: per.otto.flate@treteknisk.no

Abstract

In order to encourage increased use of wood more empirical data on the performance of wood products are needed from different exposure situations and geographical locations. In the current study modified Scots pine sapwood materials treated for above ground use were compared using: 1) two different laboratory decay tests at three different climates, 2) four different laboratory moisture tests, and 3) two different field trials. The modified wood materials in this study included acetylation, furfurylation, dimethylol dihydroxyethyleneurea (DMDHEU) and thermal modification. Copper and CCA preservatives were used as references and Scots pine sapwood as control. As expected the durability classification varied between the tests, confirming that the durability classification of a material, and the ranking between materials, is not a fixed value that can be based on one single test. Among the wood modifications acetylation had the best overall performance, the performance ranking of the other three wood modifications varied between tests. When comparing material ranking between the tests two sets of tests gave the same material ranking: 1) W 24 water vapour uptake in water saturated atmosphere and horizontal double layer above ground field exposure, and 2) mini block 11°C/70% RH and EN 252 soil contact field exposure.

Key words: *fungal decay; material resistance; moisture performance; test comparison; wood modification.*

INTRODUCTION

There is an increased awareness of the beneficial properties wood as a building material can provide. But architects, developers and engineers require documentation of reliable service life data. This causes some challenges because: 1) wood is a heterogenic material; there are variations in durability within species, 2) there are variations in performance between wood protection systems, and 3) there is no universal service life for a given species or treatment, the performance also relies on the climatic conditions and the deteriorating organisms at a given location. Hence, if a mistake is made during construction, leading to a moisture trap, the service life of a component could drastically be reduced compared to the service life expected from a well-designed and well-built construction.

It is important to keep in mind that durability is not equal to service life. Durability is a property that provides a given service life. ''The durability of wood is its resistance to wood-destroying organisms under the influence of its environment'' (Brischke *et al.* 2006). Wood species and potential wood treatment is, obviously, of great importance for the durability. In addition external factors will influence the durability, and Brischke *et al.* (2006) listed the following factors as important: 1) material temperature and wood moisture content, 2) design and execution of details, 3) conditions of use, 4) dimensions of a component, 5) cracks, and 6) the quality of workmanship. Service life is a given time period usually specified in years. The service life ends when a property reaches its critical limit of performance. Increased service life of a wood product implies less work related to replacement and lower cost for the user. It also gives a positive environmental contribution because the longer a wood product stays in use the longer it will store its carbon, and the longer one has to wait to replace a product the better since every replacement results in an environmental impact.

Unfortunately, service life prediction of wood products has so far mainly been based on expert judgement rather than empirical studies (Brischke *et al.* 2012). Brischke and Thelandersson (2014) reviewed existing modelling approaches for outdoor performance of wood products. The first comprehensive approaches include: MacKenzie *et al.* (2007), Thelandersson *et al.* (2011), Isaksson *et al*. (2014) and Meyer-Veltrup *et al.* (2017). Apart from a few comprehensive approaches using long-term field test data, service life prediction has been attempted in the past using laboratory test results. It is still controversially discussed if and to what extent durability tests under defined laboratory conditions can be used to predict the outdoor performance of wood and the effect of different environmental parameters such as different climate variables.

OBJECTIVE

The main objectives of the current study were to: 1) investigate the effect of temperature and moisture on modified wood material performance in laboratory decay trials, and 2) to compare different durability classification methods.

MATERIAL, METHOD, EQUIPMENT

Wood materials

Scots pine sapwood from the same raw material batch was used for all treatments and control specimens (SpS). This study aimed for above ground use (use class 3) treatment levels and included:

- Acetylation (Ac), weight percent gain 21, by Titanwood, The Netherlands.
- Thermal modification (TM), Thermo D, 212°C, by Scandinavian Fine Wood, Sweden.
- Furfurylation (FA), Nordic Wood Preservation Council (NWPC) class AB, by Kebony ASA, Norway. FA treatment was performed twice for field samples in order to provide full penetration.
- Dimethylol dihydroxyethyleneurea, DMDHEU (D), 1.3M, University of Göttingen, Germany.

Reference treatments included:

- Copper HDO (Cu), NWPC class AB, commercial product and treated at NIBIO.
- Copper Chromium Arsenate (CCA), NWPC class AB, commercial product and treated at NIBIO.

Laboratory decay tests

Mini block: The sample size was according to Bravery (1979), 5 × 10 × 30 (ax.) mm^3 and the test fungus was the brown rot fungus *Postia placenta* (Fr.) M.J. Larsen & Lombard (strain FPRL 280). This fungus was chosen because: 1) brown rot fungi are most relevant for conifer wood and 2) a gene expression follow up study was planned and *P. placenta* was one of the few relevant fungi with a sequenced genome. The tests were performed under three different climate conditions: 22°C/70% RH, 16°C/70% RH, or 11°C/70% RH. All materials were leached according to EN 84 (CEN 1997) prior to fungal exposure. A sterile soil medium was used containing 1/3 sandy soil and 2/3 ecological compost soil. The moisture content of the soil was adjusted to 95% of its water holding capacity according to ENV 807 (CEN 2001) and 20g sterile soil was used in each Petri dish. As inoculum a liquid culture containing 4% malt was used and 1mL was applied on each sample. The exposure time was 20 weeks and the tests were run with n = 10 replicates. The long incubation time was chosen in order to provide mass losses high enough to differentiate between materials. The weight of the plates was recorded at the start of the test and every third week. Sterile water was added when needed in order to keep the soil moisture content stable throughout the test.

ENV 807: The test was performed according to ENV 807 (CEN 2001) using specimens of 5 × 10 × 100 (ax.) mm^3 and n = 10 replicates. Deviation from the standard was that all samples were harvested after 13 weeks and that mass loss was used directly (not recalculated according to chapter A.7 in ENV 807). All materials were leached according to EN 84 (CEN 1997) prior to fungal exposure. Three different climates were used: 25°C/80% RH, 25°C/60% RH or 10°C/85% RH.

Field decay tests

EN 252: Ten replicate stakes (25 × 50 × 500 (ax.) mm³) were prepared from each wood type in accordance with EN 252 (CEN 2015). The specimens were exposed from 2010 in the NIBIO test site in Ås (the test is still running), n = 9 replicates. The stakes were assessed annually using a pick test according to the standard.

Horizontal double layer (HDL): Ten replicate stakes (25 × 50 × 500 (ax.) mm³) were prepared from each wood type. The specimens were exposed in 2009 at the NIBIO test field in Ås (the test is still running), n = 9 replicates. The specimens were placed horizontally in double layers according to Augusta (2007) with the upper layer displaced laterally by 25 mm to the lower layer. Supports were 25 cm above ground and made from aluminium L-profiles. The stakes were assessed annually using a pick test based on the EN 252 (CEN 2015) rating scheme.

Moisture tests

Ten replicate specimens of 5 × 10 × 100 (ax.) mm³ were used for three different 24 hour tests (W 24) and a capillary water uptake test.

Desorption ($W24_{0\%RH}$): Specimens were stored in 100 % RH until constant mass (approx. 2 weeks) and weighed to the nearest 0.001 g to determine mass at fibre saturation. The specimens were exposed directly on freshly activated silica gel and weighed again after 24 h. The water release of the specimens during 24 h was determined and expressed as percentage of mass at fibre saturation.

Liquid water uptake by submersion ($W24_{submersion}$): Specimens were oven-dried at 103°C until constant mass and weighed to the nearest 0.001 g to determine oven-dry mass. Specimens were submersed in a container filled with demineralised water and placed in normal climate. Specimens were separated from each other by thin spacers (cross section 1 × 1 mm²). The specimens were weighed again after 24 h submersion. The water uptake of the specimens was determined and the resulting moisture content after submersion was calculated.

Water vapour uptake in water saturated atmosphere ($W24_{100\%RH}$): Specimens were oven-dried at 103°C until constant mass and weighed to the nearest 0.001 g to determine oven-dry mass. The bottom of a miniature climate chamber (plastic container with stainless steel trays and ventilator) was filled with demineralised water. Specimens were exposed using thin spacers (cross section 1 × 1 mm²) above water in the well ventilated miniature climate chamber and weighed again after 24 h. The water uptake of the specimens was determined and the resulting moisture content after 24 h was calculated.

Capillary water uptake (CWU): Short term water absorption was measured according to modified EN 1609 (CEN 1997) procedure using a Krüss Processor Tensiometer K100MK2. Specimens of 5 × 10 × 100 (ax.) mm³ were placed in 20°C/65% RH till constant mass. The axial specimen surfaces were positioned to be in contact with water and fixed in the Tensiometer. The specimens were subsequently weighed to the nearest 0.0001 g continuously every 2 s for 200 s. The capillary water uptake was determined over time in g/cm².

RESULTS AND DISCUSSION

Laboratory decay tests

Mini block: Table 1 (first three columns) show mass losses expressed as both mean (ML_{MV}) and median (ML_{MD}). Tukey-Kramer (T-K) comparisons of ML_{MV} are provided between each material within each climate. Materials not connected by the same letter in the T-K comparison were significantly different. Among the wood modifications acetylation performed best in all climates, thermal modification, furfurylation and DMDHEU performed at statistically similar levels except for 22°C where DMDHEU had a significantly lower ML_{MV} than thermal modification. CCA had a significantly better performance than the copper HDO. It has been shown that the percentage of copper removed during leaching tests was higher from wood treated with alternative copper-based preservatives than that of CCA treated wood (e.g. Temiz *et al.* 2006). This can not fully explain the high mass loss of Cu HDO treated wood in the present study. The poor performance of the Cu HDO treated wood can potentially be due to: 1) The copper tolerant behavior of *P. placenta*. Cu-tolerance of brown rot fungi is believed to be linked to oxalic acid excretion, particularly in the initial phases of wood colonization (Clausen and Green, 2003). Oxalic acid reacts with copper in wood to form insoluble copper oxalate that is less toxic to the fungi (e.g. Humar *et al.* 2004). Boron is very prone to leaching and was likely leached from the wood during the EN 84. 2) The small sample size of the tested wood specimens. This could potentially have resulted in a very high removal of co-biocides during the EN 84 leaching procedure prior to fungal testing, predominantely boron. 3) long exposure time (20 weeks) can have increased the effect 1 (above).

Durability class (DC) categories based on ML_{MV} are indicated based on CEN/TS 15083-1 (CEN 2005a). No difference was found in durability classification between ML_{MV} and ML_{MD} or between 22°C and 16°C. Table 1, left columns, provides T-K comparison of ML_{MV} between climates within each treatment. All treatments showed significant differences between 22°C and 11°C. Only thermal modification showed significant differences between all three temperatures. A change in temperature did not drastically change the durability ranking between treatments.

Table 1

Mini block samples exposed 20 weeks to P. placenta at three different climates. Durability class categories based on CEN/TS 15083-1 (CEN 2005a); >30% DC5, >15% to ≤30% DC4, >10% to ≤15% DC3, <5 to ≤10% DC2, ≤5 DC1

	22°C/70% RH			16°C/70% RH			11°C/70% RH			Climate		
	T-K	ML_{MV} [%]	ML_{MD} [%]	T-K	ML_{MV} [%]	ML_{MD} [%]	T-K	ML_{MV} [%]	ML_{MD} [%]	22°C 70RH	16°C 70RH	11°C 70RH
SpS	A	65.5	65.7	A	65.0	65.4	A	53.4	53.2	A	A	B
Cu	A	63.3	65.6	A	53.6	57.0	B	26.3	27.4	A	A	B
TM	B	41.8	40.3	B	32.7	32.0	CD	13.7	13.5	A	B	C
FA	BC	32.4	31.4	B	31.7	31.3	BC	18.4	22.5	A	A	B
D	C	29.0	29.3	B	23.1	18.6	CDE	9.6	4.6	A	AB	B
Ac	D	1.4	1.2	C	0.1	0.0	E	0.0	0.0	A	B	B
CCA	D	0.0	0.0	C	0.2	0.0	DE	1.4	0.1	B	AB	A

ENV 807 (CEN 2001): Table 2 (first three columns) provides mass losses expressed as both ML_{MV} and ML_{MD} and T-K comparison of ML_{MV} between each material within each climate. Wood modifications had less than 3% mass loss in all climates. Acetylation and thermal modification performed significantly better than furfurylation in all climates. Mass loss categories are indicated based on CEN/TS 15083-1 (CEN 2005a). No difference was found in DC classification for the modified wood materials in this test. With longer exposure time the differences in material performances, i.e. mass loss, would have been clearer. Table 2, left columns, provides T-K comparison of ML_{MV} between climates within each treatment. For the materials with high mass loss (Scots pine sapwood, copper HDO and CCA) a significant difference was found between 65% RH and 85% RH. Due to the low mass loss of the treated materials in this test the comparisons between materials and climates are only indicative. In an ENV 807 test by Westin and Alfredsen (2007) modified wood performed better than both copper chromium and CCA preservatives in compost soil after 40 weeks incubation.

Table 2

ENV 807 samples exposed 13 weeks at three different climates. Durability class categories based on CEN/TS 15083-1 (CEN 2005a); >10% to ≤15% DC3, <5 to ≤10% DC2, ≤5 DC1

	25°C/80% RH			25°C/60% RH			10°C/85% RH			Climate		
	T-K	ML_{MV} [%]	ML_{MD} [%]	T-K	ML_{MV} [%]	ML_{MD} [%]	T-K	ML_{MV} [%]	ML_{MD} [%]	25°C 85RH	25°C 65RH	10°C 85RH
SpS	A	12.0	12.7	A	9.2	9.5	A	2.7	2.2	A	B	C
Cu	B	4.6	4.3	BC	1.7	0.6	BC	1.3	1.3	A	B	B
CCA	BC	3.1	2.9	BC	1.2	0.8	B	1.4	1.4	A	B	B
FA	CD	2.6	2.4	B	2.6	2.6	AB	1.8	1.9	A	A	B
D	DE	1.1	1.3	BC	1.0	1.0	B	1.4	1.2	A	A	A
TM	E	0.1	0.0	C	0.0	0.0	CD	0.4	0.3	B	B	A
Ac	E	0.0	0.0	C	0.0	0.0	D	0.0	0.0	A	A	A

When comparing the modified wood results from the sterile mini blocks exposed to *P. placenta* with the unsterile ENV 807 samples exposed to a mixed community of soil inhabiting fungi the most striking difference was for thermal modification. In the mini block test the highest mass loss among the wood modifications was found for thermal modification, and there were no significant differences between thermal modification and furfurylated wood. In the ENV 807 test only initial signs of decay were detected in thermal modification and no significant difference was found between the performance of thermal modification and acetylation. The explanation might simply be the low mass losses, or there might be an effect due to different wood degrading organisms.

Field decay tests

EN 252: The data for soil contact exposure in Table 3 show that Scots pine sapwood had the most severe decay and acetylation the least severe decay after six years of exposure. No significant difference in performance was found between the reminding treatments. The EN 252 (CEN 2015) standard states that: ''*The test shall be run for a minimum period of five years (or until all stakes have failed if this occurs earlier). It is advisable to continue the test beyond five years, with inspections at suitable intervals, in order to determine longer performance of the treated stakes. NOTE: Ideally the test should be continued until all stakes of the product under test have failed*.'' The latter is the only way to determine the actual average service life of the tested materials in the respective test.

HDL: Table 3 also illustrates that Scots pine sapwood had the most severe decay after seven years of exposure in HDL. The treated samples fell into two groups based on the T-K comparison: 1) DMDHEU, thermal modification and copper HDO with a mean decay rating between 1.6 and 2.1 after seven years and 2) furfurylation, acetylation and CCA with only initial signs of decay, mean decay rating of 0.2 or below. The HDL is believed to be comparable to worst case scenario for wood in service above ground, i.e. a badly constructed detail with a moisture trap. The HDL is a non-standard test and no guidance is given with respect to how long the test should run. To keep samples in the field trial until all stakes fail in order to provide actual service life data is a good recommendation also for the HDL test.

Table 3

Mean and median decay rating of treated wood in above ground HDL after seven years and EN 252 (CEN 2015) after six years

	EN 252			HDL		
	T-K	**Mean decay**	**Median decay**	**T-K**	**Mean decay**	**Median decay**
SpS	A	3.0	4.0	A	2.6	3.0
D	B	2.2	3.0	AB	2.1	2.0
TM	B	2.6	3.0	B	1.7	2.0
Cu	B	2.6	2.0	B	1.6	2.0
CCA	B	1.9	2.0	C	0.2	0.0
FA	AB	2.8	3.0	C	0.1	0.0
Ac	C	0.0	0.0	C	0.1	0.0

The major difference between above ground and soil contact testing is believed to be higher and more stable moisture content in the wood samples provided by soil contact exposure and direct contact to soil-inhabiting decay organisms. For modified wood the most obvious difference between the two tests are how furfurylated wood only had initial decay in HDL, but when exposed in soil contact no significant difference was found between furfurylation and Scots pine sapwood. Results from long term field trials on modified wood are still sparse, but Larsson-Brelid *et al.* (2010) found that after 18 years in soil contact wood with acetyl content of about 20% was of the same magnitude as for CCA treated wood at a high retention level (10.3kg m^{-3}).

It is important to remember that the pick test rating normally used for field trials is not very sensitive, it can suffer from some unintended evaluator bias, and the decay rating scale is not linear. Based on experience from Norwegian field trials samples can be rated 1 for a long time due to surface softening. Hence, one should be careful comparing materials with initial decay (i.e. rating 2 or below). In addition, if the degradation starts in the central part of a specimen the decay might not be detected with the pick test. This is predominately the case with copper treated wood (Humar and Thaler 2017).

Moisture tests

Table 4 provides the data from the 24 hour tests and capillary water uptake. For modified wood reduced water affinity is believed to be of great importance when it comes to mode of action mechanisms against wood deteriorating organisms while wood preservatives are mainly relying on a toxic effect (Hill 2006). This is also reflected in the results below, generally modified wood had lower moisture uptake than the preservative treated wood and untreated control.

$W24_{0\%RH}$: The highest values for desorption were determined for Scots pine sapwood, the lowest for acetylation. Acetylation had significantly lower desorption values than DMDHEU and furfurylation. No significant difference was found between DMDHEU and furfurylation or between thermal modification and acetylation.

$W24_{submersion}$: The highest value for liquid water uptake by submersion were found for Scots pine sapwood, the lowest for furfurylation. No significant difference was found between acetylation and DMDHEU or between thermal modification and DMDHEU.

$W24_{100\%RH}$: For water vapour uptake in water saturated atmosphere the highest value were found for copper HDO, the lowest for acetylation. No statistically significant differences were found between acetylation, furfurylation and thermal modification or between DMDHEU and thermal modification.

CWU: Capillary water uptake showed the highest value for Scots pine sapwood and lowest value for furfurylation. No significant difference was found between acetylation, thermal modification and DMDHEU or between furfurylation, thermal modification and DMDHEU.

Table 4

W 24 tests and capillary water uptake

	$W24_{0\%RH}$		$W24_{submersion}$		$W24_{100\%RH}$		CWU	
	T-K	**Mean [%]**	**T-K**	**Mean [%]**	**T-K**	**Mean [%]**	**T-K**	**Mean [g/cm²]**
SpS	A	17.2	A	93.2	ABC	17.2	A	0.34
Cu	ABC	13.7	B	79.2	A	24.1	B	0.18
CCA	ABC	14.6	CD	54.5	AB	23.3	C	0.13
D	AB	15.9	CD	52.2	BC	15.9	CD	0.11
FA	BC	12.3	E	23.6	D	7.6	D	0.09
TM	CD	10.2	C	60.6	CD	13.4	CD	0.11
Ac	D	6.5	D	45.0	D	6.5	C	0.12

In Table 5 x-values and durability classes based on the CEN/TS 15083-2 (CEN 2005b) approach are provided for all decay tests, i.e. the x-value is ML_{MD} of wood test species divided by ML_{MD} of reference test specimens (Scots pine sapwood). For the field trials decay rating was used instead of mass loss. This way of comparing different tests is stretching the original methodology, especially since the four decay rating categories are broad and the evaluation method not very sensitive plus that it not nesessarily is a good linear relation between the four decay rating categories. Hence, this table is a rough way to compare all decay tests in this study.

The only material with the same DC ranking in all decay tests is acetylation (DC 1). When summing up, for each treatment, the DC that occured at least three times: acetylation DC 1, CCA DC1, DMDHEU DC 2, furfurylation DC 3-4, thermal modification DC 4 and copper HDO DC 4. It is important to keep in mind that above ground retentions were used also in soil contact field trials.

Table 5

The x-value and durability class (in shades of grey) are given using Scots pine sapwood as reference species. The x values and durability classes according to CEN/TS 15083-2 (CEN 2005b); >0.80 DC5, >0.45 to ≤0.80 DC4, >0.20 to ≤0.45 DC3, >0.10 to ≤0.20% DC2, ≤0.10 DC1.

	Mini block						ENV 807						HDL		EN 252	
	22°C 70RH		16°C 70RH		11°C 70RH		25°C 80RH		25°C 60RH		10°C 85RH					
	x value	DC	x value	DC	x value	DC	x value	DC	x value	DC	x value	DC	x value	DC	x value	DC
Ac	0.0	1	0.0	1	0.0	1	0.0	1	0.0	1	0.0	1	0.0	1	0.0	1
D	0.4	3	0.3	3	0.1	2	0.1	2	0.1	2	0.5	4	0.7	4	0.8	5
FA	0.5	4	0.5	4	0.4	3	0.2	3	0.3	3	0.8	5	0.0	1	0.7	4
TM	0.6	4	0.5	4	0.3	3	0.0	1	0.0	1	0.1	2	0.7	4	0.8	5
Cu	1.0	5	0.9	5	0.5	4	0.3	3	0.1	2	0.6	4	0.7	4	0.5	4
CCA	0.0	1	0.0	1	0.0	1	0.2	2	0.1	2	0.7	4	0.0	1	0.5	4

In Table 6 the ranking of materials from all the different decay and moisture tests, based on ML_{MV}, decay rating or moisture performance, is provided. The highest performance within each test is given the value 1, the second best value 2, the third best value 3 and the lowest value 4. Then the sum from all tests was calculated for each material. The overall best performance was achieved for

acetylation (score 16), while thermal modification (score 34), DMDHEU (score 34) and furfurylation (score 35) perfomed at a similar level in these tests.

The difference in material perfomance ranking between tests were also compared. Tests with similar ranking included: 1) W $24_{100\%\ RH}$ and HDL, and 2) mini block 11°C/70% RH and EN 252. The comparison also showed no difference between the three climates in the ENV 807 test or between 22°C and 16°C in the mini block test. The material ranking for CWU, $W24_{0\%RH}$ and $W24_{submersion}$ did not overlap with any of the other tests.

Table 6

Ranking of treatments from all tests, lowest performance (i.e. highest mean mass loss, decay rating or water uptake) to the left, highest performance (i.e. lowest mean mass loss, decay rating or water uptake) to the right.

	Ranking low ⟶ high			
Mini block 22°C/70%	TM	FA	D	Ac
Mini block 16°C/70%	TM	FA	D	Ac
Mini block 11°C/70%	FA	TM	D	Ac
ENV 807 25°C/80%	FA	D	TM	Ac
ENV 807 25°C/60%	FA	D	TM	Ac
ENV 807 10°C/85%	FA	D	TM	Ac
$W24_{0\%RH}$	D	FA	TM	Ac
$W24_{submersion}$	TM	D	Ac	FA
$W24_{100\%RH}$	D	TM	FA	Ac
CWU	Ac	D and TM		FA
HDL	D	TM	FA	Ac
EN 252	FA	TM	D	Ac

CONCLUSIONS

In the current study of UC3 retentions of modified Scots pine sapwood in different laboratory moisture and decay tests and two field trials acetylation provided the best overall performance. The durability performance of thermal modification, furfurylation and DMDHEU varied between the different tests, confirming that the durability classification of a material, and the ranking between materials, is not a fixed value that can be based on one single test.

When comparing material ranking between tests W 24 water vapour uptake in water saturated atmosphere and HDL + mini block 11°C/70% RH and EN 252 gave similar rankings.

The W24 test (i.e. moisture response) showed that the modified wood, as expected, had lower moisture uptake than the preservative treated wood and the control. However, the moisture behaviour could not fully explain the decay resistance of the materials.

Lowering the temperature did, as expected, slow down the decay rate, but it did not make much difference in the durability ranking of the materials.

In order to predict service life the authors recommend to combine decay and moisture data, e.g. as in Meyer-Veltrup *et al.* (2017). This is especially important for modified and preservative treated wood since different modes of action need to be considered.

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HOW DO BIOTIC AND ABIOTIC FACTORS COMBINE TO AFFECT THE WEATHERING OF WOOD SURFACES?

Julia BUCHNER
Ecole Supériere du Bois, LIMBHA, Rue Christian Pauc, 44306 Nantes, France
Tel: 0033 2 40 18 12 12, Fax: 0033 2 40 18 12 00, E-mail: julia.buchner@ecoledubois.fr

Mark IRLE
Ecole Supériere du Bois, LIMBHA, Rue Christian Pauc, 44306 Nantes, France
Tel: 0033 2 40 18 12 12, Fax: 0033 2 40 18 12 00, E-mail: mark.irle@ecoledubois.fr

Christophe BELLONCLE
Ecole Supériere du Bois, LIMBHA, Rue Christian Pauc, 44306 Nantes, France
Tel: 0033 2 40 18 12 12, Fax: 0033 2 40 18 12 00, E-mail: christophe.belloncle@ecoledubois.fr

Franck MICHAUD
Ecole Supériere du Bois, LIMBHA, Rue Christian Pauc, 44306 Nantes, France
Tel: 0033 2 40 18 12 12, Fax: 0033 2 40 18 12 00, E-mail: franck.michaud@ecoledubois.fr

Nicola MACCHIONI
IVALSA, Via Madonna del Piano 10, 50019 Sesto Fiorentino, Italy
Tel: +39 55 52 25 502, Fax: +39 55 52 25 507, E-mail: macchioni@ivalsa.cnr.it

Abstract

The paper presents the aim and objectives of a research project concerned with the weathering processes of oak (Quercus robur.) and douglas fir (Pseudotsuga menziesii). The project deals with the identification of any synergistic effects between biotic factors such as aerobic bacteria, Basidiomycota, Ascomycota and abiotic factors such as light, temperature and moisture on the weathering of wood surfaces in durability class 3.

Key words: *abiotic factors; fungi; bacteria; artificial accelerated weathering; outdoor weathering.*

INTRODUCTION

Exterior wood applications are used for example in the building industry, for outdoor furniture and various wooden structures. When wood is used as a facade material it changes colour, cracks and rots after several years in service. In general, customers seek materials that need low maintenance and retain a homogeneous surface for a long period of time. Nevertheless, not only wood but also concrete, metal, glass and plaster are in need of cleaning, repainting and repairing after several years in service. It is proposed that if economic methods that minimise the maintenance required of wood then this environmental friendly material would be used more often. The use of wood as a renewable raw material is normally considered to be CO_2 neutral and more ecological than some of the materials mentioned above, as shown in a life cycle assessment by Salazar and Meil (2009). According to Börjesson and Gustavsson (2000), where the life cycle assessment of a wood building construction, was compared to a concrete building, the use of wood products can reduce the greenhouse gas emissions drastically.

Wood, like all materials, is degraded by abiotic factors, e.g. UV light, hydrolysis, physical erosion, etc. In addition, because it is an organic material, it can also be susceptible to biological degradation, i.e. biotic factors, especially by micro-organisms. This project will investigate the existence of any synergistic effects between biotic and abiotic factors during the weathering of wood surfaces. If a better understanding of which factors affect wood in which manner and in which combination biotic and abiotic factors interact to degrade the wood, it might be possible to develop techniques that increase the life-span of wood, but, which have minimal environmental impacts. This approach will facilitate the design of stable wooden facades that require little maintenance over a long period of time.

DEGRADATION OF NATURAL WOOD

Extensive research on the decay of wood in water or soil contact has already been carried out. There is a lack of knowledge concerning the degradation process of wood in end-use class 3 through

microorganisms but also through abiotic factors such as light, temperature and moisture and especially the interaction of all factors.

Research on bacterial communities on sunken wood in the Mediterranean Sea revealed that in an anaerobic environment, a high diversity of microbial communities exist (Fagervold et al. 2014). Most likely in anaerobic as well as in aerobic environments the diversity of microbial communities is very high.

The wood degrading action bacteria, and thus aerobic bacteria have been studied for example by Daniel (1994) and are reported to degrade lignocellulose preparations with the help of the lignin peroxidases enzyme. Schmidt and Liese (1994) mention however, that only few bacteria show activity towards lignin. Schmitz (1919) reported first that a mix of bacteria and fungi populations caused a greater deterioration in wood compared to decay by the basidiomycetes alone. He moreover found that some bacteria produce ammonia which increases the pH value. Hervé et al. (2016) found by conducting a microcosm experiment, that fungal growth was significantly lower when soil bacteria was present. These changes on the growth environment caused by bacteria may have an influence on the ability of fungal development. Bacteria appear primarily with parenchymatous tissues and thus they often accumulate in rays and resin ducts. Moreover an affinity towards the S3 layer in softwood tracheids and hardwood fibres and vessels could be detected (Wilcox 1970). According to multiple studies such as Greaves (1969), Eriksson et al. (1990) and Fengel and Wegener (1989) there are three different kind of attacks on pit borders caused by bacteria: Tunnelling, erosion and cavitation. These researches make it particularly interesting to situate the following hypothesis: Bacteria, deriving in an aerobic environment have an influence on the degradation process of wood without ground or direct water contact.

The variation of the surface wetting is very different from one wood species to another and plays an important role in the degradation process of wood. Thus the sample size, orientation and alignment of the weathering samples have to be considered carefully. Studying the surface wetting of different wood species during weathering, Oberhofnerová and Pánek (2016) found a large fall in the contact angle between 6 and 12 months. In a study by Oberhofnerová et al. (2017) on the effects of natural weathering of wood, the roughness, colour and the formation of cracks changed more rapidly during the first months of weathering. According to George et al. (2005) the chromatic coordinates of irradiated oak sapwood and heartwood evolve in opposite directions after only short irradiation times. Pursuant to these findings the process of wood weathering is very fast at the beginning and thus the degradation process has to be monitored more precisely during the initial stages.

The aim of this thesis is to identify any synergistic effects between biotic factors, such as bacteria, fungi and moulds as well as abiotic factors, such as light, temperature and moisture on the weathering of wood surfaces in durability class 3.

OBJECTIVES

Accelerated and outdoor weathering experiments will be carried out to see if and how environmental conditions and in particular, the presence of micro-organisms, influence the weathering of wood.

One of the research objectives is to study the interactions between abiotic and biotic factors in a laboratory environment and therefore an artificial accelerated weathering experiment will be conducted. A study by Kataoke et al. (2007) shows that the depth of photodegradation is greater than indicated by other researchers such as Hon and Chang (1984), because visible light was found to penetrate 540 µm below the surface. Moreover the study shows that blue light can cause bleaching of the wood. Due to these findings and the fact that UV light is known to be an effective disinfectant, for this artificial accelerated weathering test the light source which is proposed to use by standards such as ASTM G0154 (2016) and ISO 16474-1 (2014) will be modified.

Another objective is to study the interactions between abiotic and biotic factors in natural conditions. An outdoor weathering experiment will be conducted in western France as well as in Italy. Meteorological data will be precisely recorded, consequently the degradation process of wood in two different regions can be compared to each other. This realistic experiment will allow researchers but also industry to understand the degradation process of the specific wood species.

Microbiological and molecular biology methods will be used to identify and quantify bacteria, moulds and fungi present in degraded wood facades in western France and Italy. A microcosm-scale experiment may reveal if certain bacteria have an influence on the growth rate of certain fungi and moulds or vice versa.

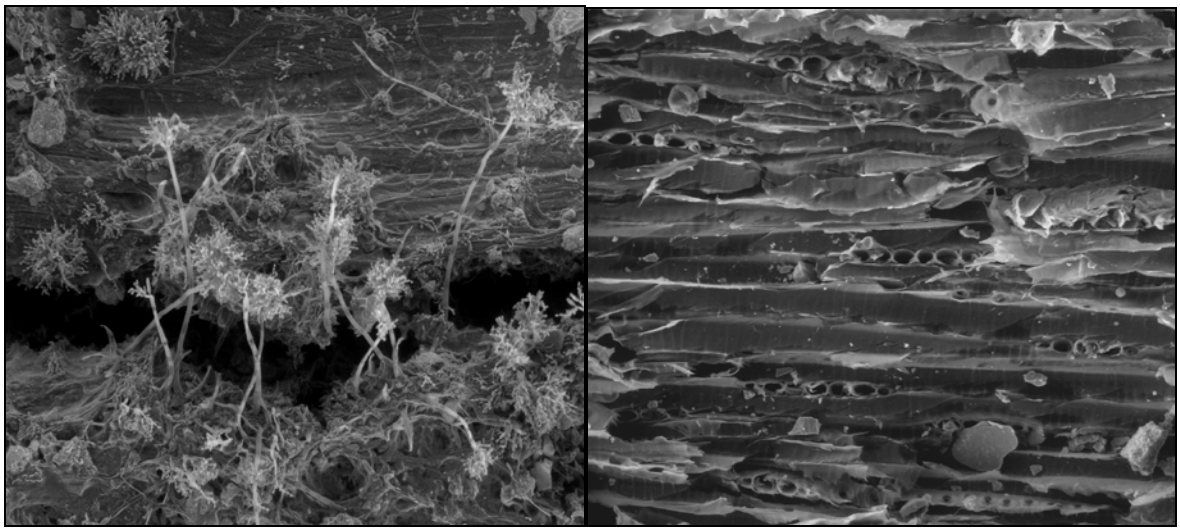

Fig. 1.
Left: Oak wood stored in water for 4 days, fungi development. Right: Douglas fir wood naturally weathered for 4 weeks.

MATERIAL AND METHODS

Douglas fir (*Pseudotsuga menziesii*) and oak (*Quercus robur*) will be examined due to the fact that these two species are commonly used for exterior applications in Europe. Moreover this choice allows the comparison between softwood and hardwood.

Several methods will be used to analyse the anatomical (ESEM, light microscope), chemical (FTIR/ATR), and visual (colorimeter, roughness testing) changes in the degraded wood as well as a surface contact test, counting the CFU will help to classify the fungi and bacteria. The focus however is on the change in visual appearance of the weathered wood surface.

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INFLUENCES OF SOME FACTORS ON ADHESION STRENGTH BETWEEN PVC FOIL AND PARTICLE BOARD

Dimitar ANGELSKI
University of Forestry, Faculty of Forest Industry
10 Kliment Ohridsky Blvd., 1756 Sofia, Bulgaria
Tel: 00359 887 567168, Fax: 00359 2862 28 30, E-mail: d.angelski@ltu.bg

Pavlin VITCHEV
University of Forestry, Faculty of Forest Industry
10 Kliment Ohridsky Blvd., 1756 Sofia, Bulgaria
E-mail: p_vitchev@ltu.bg

Vladimir MIHAILOV
University of Forestry, Faculty of Forest Industry
10 Kliment Ohridsky Blvd., 1756 Sofia, Bulgaria
E-mail: v.mihailov@ltu.bg

Abstract

The article discusses the influence of some technological factors on the through feed wrapping of wood-board materials with foils. For the aim of the study particle board details with dimensions 350/50/18 mm have been used. The specimen details were wrapped through feed at an automatic panel wrapping machine "FUX Austria" with polyvinyl chloride (PVC) foil. The used materials in the experimental operation were as follow: particle board from „Kronospan" company and PVC foil made by „Hornshuch". The wrapping of the specimen details was made with reactive hot melt polyurethane glue Purmelt QR 5300 from „Henkel"-Germany. The main characteristic of the adhesive connection between the materials is its adhesion strength. In this relation, the tensile strengths of the compound has been determined, perpendicular to the plane of adhesion. The influence of the following technological factors on the adhesion strength was also investigated: temperature of glue, quantity of glue, feeding speed. Three-factor experiment has been accomplished at three levels of the factors values. The adhesion strength has been determined following the RAL RG 716/1 standard, Part 7, according to DIN 16 860. The experimental data were processed using the regression analyses method and a mathematic equation has been done to determine the relationship interrelation between the factors and the output data value. The dependencies are presented by graphics and the results are analyzed.

Key words: *particle board; PVC foil; adhesion strength; technology factors of laminating.*

INTRODUCTION

In furniture made of wood and wood-based materials, the technology of bonding of multiple thin layers of veneers is most commonly used. The adhesive material aims to ensure a rigid, irreversible and sufficiently strong fixation of the laminating material to the elaborated surface. The surface of the furniture elements to be laminated, should be smooth, homogenous and clean out of dust and other contaminants. The medium-density fiberboards (MDF) as well as some multilayer high quality particleboards easily met these requirements. Lamination in the furniture production is performed either stationary or through feed. In the recent years, the through feed laminating technologies draw a lot of attention and are widely used in the modern furniture plants with large-volume production.

Polyvinylchloride (PVC) is one of the most flexible and durable laminating material. Thus, the PVC foil is preferred for the through feed wrapping of furniture elements. One of the main technological disadvantages in lamination with foils is that usually overlays are very thin and they do not have the strength to hide the surface irregularities of the laminated article. This defect known as "marking" negatively affects the adhesion strength of the bonding component (Kılıç et al. 2009). Knowing this, the panels need to be flat with a maximal deviation in the thickness of ±0,2mm and to lack imperfections which could be "marked" through the foil. Since the foils do not absorb water and steam, another important requirement for the panels is to possess even minimal water and steam penetration to render uptake of the glue solvents. In order to achieve minimal shrinkage of the adhesive layer during its hardening, it is recommended the adhesives to have the highest possible dry residue content (Albin et al. 1991).

For through feed wrapping with PVC foil, polyurethane (PUR) hotmelt glues are preferred. Their benefits include immediate bonding, no solvents are used, water resistance and low application temperature that prevents distortion of heat-sensitive substances. Depending on air humidity and material conditions the polyurethane hotmelt adhesives can achieve 50-80% of the ultimate bond strength in approximately 6 hours with a full cure achieved in 24 hours. The main parameter for the quality of the wrapping process of the furniture panels is the adhesive strength of the bonding material. Adhesion is a complex physico-chemical phenomenon for which, however, there is not a rigorous theoretical definition (Kaelblea 1964, Silva et al. 2011). One of the most common and accepted techniques for measuring polymer adhesion is the peel test (Zosel 1991). The method is simple and represents one of the main problems of the laminated furniture elements - delamination. This is grounds for the wide use of this method in the assessment of the final product. Basic technological parameter of the through feed wrapping process is the feeding speed of the details. For the laminating process using PVC foil the feeding speed is recommended to be between 6 to 15m/mm (Albin et al. 1991). It is well-known that the adhesion is influenced by the temperature and the quantity of the applied adhesive. The values of these parameters depend on the glue type. The smooth panel surface, accompanied by its lower permeability requires smaller amount of the adhesive. When high quality adhesive material and furniture panels meeting all the above mentioned requirements are used, the adhesive strength should depend mainly on the parameters of the coating process. The influence of some fundamental technical parameters on the coating quality could be evaluated through derivation of a regressive equation.

OBJECTIVE

The aim of the current study was to evaluate and compare the influence of the fundamental techical parameters: speed feed (U), quantity of the adhesive material (Q), temperature of the adhesive material (t_g) on the through feed wrapping of particle boards with PVC foil. This study is a follow up of our previous work where we determined the influence of the above-mentioned parameters on the adhesive strenght over a through feeding wrapping process of MDF with PVC foil (Angelski and Vitchev 2014). For the assessment, a standartzed peeling test was performed. An additional objective of this sudy was to evaluate the influence of the basic material (board for lamination) on the strength of the adhesive material.

MATERIAL, METHOD, EQUIPMENT

Materials

For the ojectives of the study, particle boards with thickness of τ 680kg/m^3 have been used. A total of 75 samples (50/300/18mm) have been made. Semi-rigid PVC foil, UV-protected by transparent acrylic film with a thickness of 0,18÷0,22mm was used as coating material.

Reactive hotmelt polyuretane adhesive system "Purmelt QR 5300" (Henkel) was chosed as a bonding material. Purmelt QR 5300 had viscosiy of 26000mPa s/130°C and melting point about 65°C. The operating temperature, recommended by the manufacturer is between 110°C to 150°C and the quantity of the applied glue (Q) should be between 40 to 100g/m^2.

Experimental Method

To determine the influence of the selected technical parameters on the adhesive strength of the adhesive substance, a method of regression analysis was used. As it is known, the changes of the output value depending on the variation of the values of the technical factors could be expressed by a parabolic regression equation of second order. On this basis, a matrix composition plan of G.Box (Box et al. 1951) strongly influencing the adhesion technical factors has been designed and performed.

The factors with lower influence on the adhesion have constant values, considered beneficial for the adhesive process. The variable factors vary at three levels: maximum, medium and minimum. For convenience of the mathematical analysis of the data, the factors in the experimental matrix are given with the following codes: maximum (+1); medium (0); minimum (-1). In the carried out experiment the values of the variable factors in non-coded form are as follows:

- quantity of the applied adhesive material Q (x_1) – 60, 80, 100g/m2;
- temperature of the applied adhesive material t (x_2) – 110, 135, 160°C;
- feeding speed U (x_3) – 6, 13, 20m/min.

The through feeding lamination of the tested article has been performed on a specialized automated profile laminating machine „FUX Austria". The working temperature of the applied adhesive material and the feeding speed are set automatically by the used software. The quantity of the adhesive to be applied is calculated on the basis of the foil width using a mathematical algorithm. The polyurethane hotmelt adhesive is applied on the PVC foil seconds before coating of the elements.

Passing through the coating zone, silicone-pressing rollers are shaping the foil onto the element. The results are expressed as averaged values of five experiments. Statistical programme "Qstatlab 5" was used for the analysis of the data and calculation of the regression coefficients.

Test method

The adhesion has been determined by peel test based on the standard RAL RG 716/1 according DIN 16 860, Part 7. The samples were tested 24 hours after coating. The preparation includes stationary positioning of the samples and cutting the foil. The metal former was positioned over the foiled side of the test sample and two cuts with knife have been made upon it so that lines with 36 mm width to be cut. Afterwards, the edge of the cut foiled line was unstuck and gripped by a mechanical pulling device and subsequently peeled away from the particle board at a constant speed and angle of peeling (Fig. 1). An electronic digital weight scale was attached to the mechanical pulling device. During the test, the peeling speed and peeling force were recorded. The recorded destructive forces were re-calculated and expressed in N/mm. On the basis of these data the influence of the assessed factors on the adhesive strength of the adhesive components has been evaluated.

Fig. 1.
Peeling test.

RESULTS AND DISCUSSION

Matrix compositional plan and average values (*F*p) obtained from the peeling test are shown in Table 1. The second order equation from which the regression coefficients have been derived is as follow:

$$y = 1{,}53 + 0{,}34\,x_1 + 0{,}34x_2 - 0{,}37x_3 + 0{,}05x_1x_2 - 0{,}03x_1x_3 + 0{,}14x_2x_3 - 0{,}01x_1^2 - 0{,}1x_2^2 + 0{,}07x_3^2 \quad (1)$$

The influence of the technological factors on the strength of the adhesive component is presented in Fig. 2, 3 and 4. From the results, it is visible that the adhesion strength of the adhesive material does not exceed 2N/mm.

Table 1

Matrix compositional plan and average values from the peel test

№	$Q \equiv x_1$ g/m^2	$t \equiv x_2$ °C	$U \equiv x_3$ m/min	Fp_1 N/mm	Fp_2 N/mm	Fp_3 N/mm	Fp_4 N/mm	Fp_5 N/mm	Fp N/mm
1	60(-)	110(-)	6(-)	1.455	1.305	1.175	1.490	1.320	1.349
2	60(-)	110(-)	20(+)	0.615	0.430	0.460	0.480	0.535	0.504
3	60(-)	160(+)	6(-)	1.655	1.580	1.655	1.840	1.710	1.688
4	60(-)	160(+)	20(+)	1.235	1.310	1.215	1.430	1.290	1.296
5	100(+)	110(-)	6(-)	1.900	1.965	1.985	1.890	1.940	1.936
6	100(+)	110(-)	20(+)	0.895	0.895	0.840	0.795	0.830	0.851
7	100(+)	160(+)	6(-)	2.165	2.350	2.200	2.630	2.360	2.341
8	100(+)	160(+)	20(+)	1.980	1.795	1.990	2.155	1.905	1.965
9	100(+)	130(0)	13(0)	1.840	1.995	2.240	2.135	2.240	2.09
10	60(-)	130(0)	13(0)	0.835	0.900	0.990	0.970	1.145	0.968
11	80(0)	160(+)	13(0)	1.880	2.040	2.120	1.900	1.840	1.956
12	80(0)	110(-)	13(0)	0.845	0.840	0.765	0.960	1.090	0.900
13	80(0)	130(0)	20(+)	0.980	1.105	1.110	1.080	1.065	1.068
14	80(0)	130(0)	6(-)	2.280	2.180	1.845	2.090	2.230	2.125
15	80(0)	130(0)	13(0)	1.340	1.360	1.235	1.330	1.275	1.308

According to the DIN 16 860 standard the adhesion strength is recommended to be over 2,5N/mm. The tested samples do not meet this requirement. On the other hand their adhesion strength is by 0,7N/mm lower compared to the strength of the adhesives used for bonding the PVC foil to the MDF, using the same polyurethane adhesive (Angelski and Vitchev 2014).

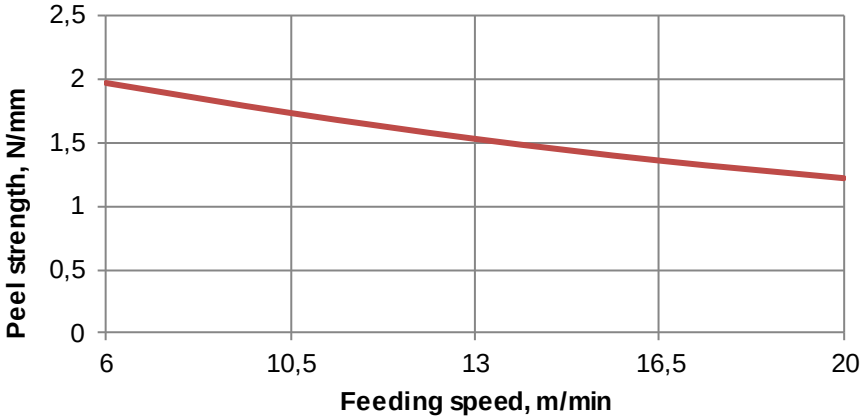

Fig. 2.
Relationship between peel strength of glue joint and feeding speed during through feed lamination of particle board with PVC foils.

In principle, the bonding strength of the adhesive components could be increased by improving the quality of the surface subjected to lamination. Improving the smoothness of the surface of particleboards can be effectively achieved by performing the following technological operations: coating a special plaster, drying of plaster and sanding. These processes, on the other hand would increase the duration of the technical cycle and the production price. Based on these data we could draw the conclusion that the subjected to coating with PVC foil furniture elements are preferable to be made out of MDF instead of particleboards.

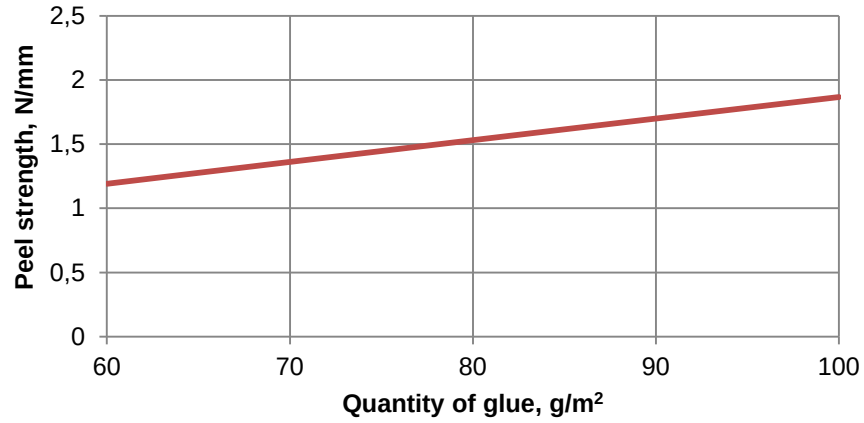

Fig. 3.
Relationship between peel strength of glue joint and quantity of glue during through feed lamination of particle board with PVC foil.

The regression coefficients in front of the variable factors x_1, x_2 and x_3 in the derived equation have approximately equal values. This means that the variable technological factors exert commensurable influence on the adhesion strength of the adhesive component. Their variations in the test range could result in either an increase or a decrease by 30% of the adhesion strength of the adhesive. The low values of the regression coefficients determine the weak parabolic relationship presented in Fig. 1, 3 and 4. There is a dual interaction between the feeding speed and the temperature of the adhesive component on the adhesion strength. This means that the combination of high temperature of the adhesive with high feeding speed should be avoided. The inversely

proportional relationship between the feeding speed (*U*) and the adhesion strength of the adhesive component is clearly depicted in Fig. 2. In order to produce objects with high strength the recommended feeding speed in coating is between 6 to 10m/min.

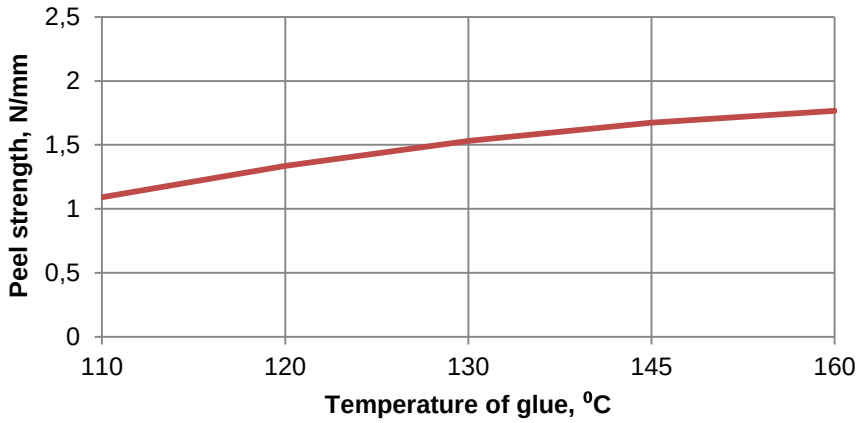

Fig. 4.
Relationship between peel strength of glue joint and temperature of glue during through feed lamination of particle board with PVC foil.

After the optimization performed by the scanning method the highest adhesive strength value of the adhesive component was achieved at *Q* = 100 (g/mm), *t* = 160 (^{0}C), *U* = 6 (m/min). Higher adhesive strength could be achieved and by increasing the adhesive quantity (Fig. 3). This, however is regarded as unfavorable to the technological process, since a larger amount of adhesive material would lead to smearing the surface as well as the polyurethane hotmelt adhesives are relatively expensive.

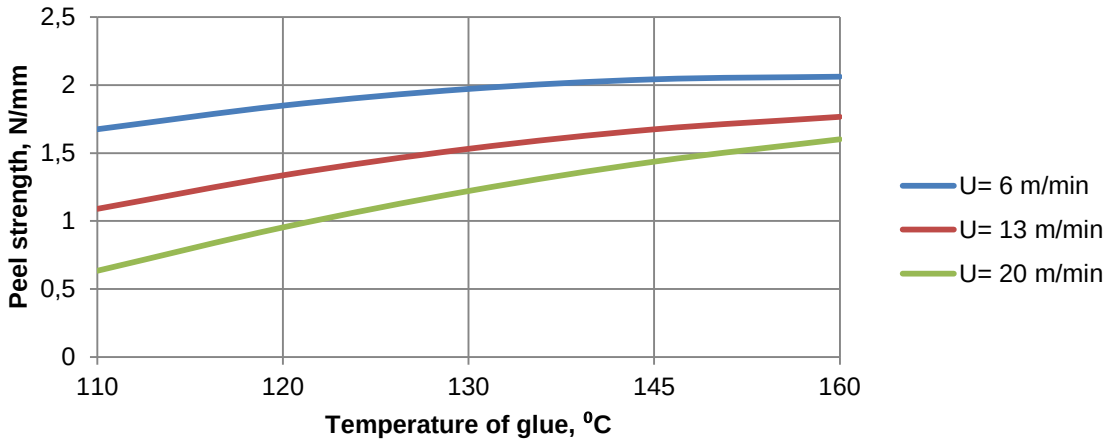

Fig. 5.
Relationship between peel strength of glue joint, feeding speed and temperature of glue during through feed lamination of particle board with PVC foil.

The range of the temperature variations of the melt is restricted as by the working viscosity of the adhesive component, as well as by the temperature at which the adhesive is destructed. Generally, the bonds formed at higher temperatures of the adhesive component possess also higher adhesion strength (Fig. 4). This means that in the lack of restricted conditions higher temperatures are preferred. Even though, as it is already mentioned above, the technological relationship between the temperature of the adhesive component and the feeding speed. At high feeding speed the cooling time of the hot melt adhesive may not be sufficient and may hamper the following process of removing the protruded parts of the PVC foil. Thus, the thermal conductivity of the material has to be taken into account when the through feed wrapping process is set up. In relation to this, the combined influence

of these two factors - temperature and feeding speed on the adhesion strength of the adhesive component is shown in Fig. 5. The most favorable conditions for through feeding wrapping of particles boards are achieved at feeding speed over 10m/min and temperature of the adhesive component below 130^0C. This is due to the fact, that at higher feeding speed the compression is insufficient for obtaining of high quality adhesive components.

CONCLUSIONS

On the basis of our study and the results obtained, the following conclusions regarding the feed through lamination of particle boards with PVC foil and polyurethane hotmelt adhesive could be drawn:

- The adhesive components possess lower adhesion strength compared to those, recommended by the DIN 16 860 standard (adhesion strength over 2,5N/mm). In order to obtain adhesives with the recommended adhesion strength, the smoothness of the subjected to lamination article should be increased. Technologically, this can be achieved by plastering the particleboard surface with subsequent drying and sanding. In our previous study, we found that the standard requirements are met when the coated articles are MDF. Considering this, we conclude that the subjected to coating with PVC foil furniture elements are preferable to be made out of MDF instead of particleboards.
- The three evaluated technological factors: feeding speed (U), quantity (Q) and temperature (t) of the applied adhesive material exert commensurable influence on the adhesion strength of the adhesive component. Their variations in the tested range could result in either an increase or a decrease by 30% of the adhesion strength of the adhesive.
- For the tested range of the factors variation, the highest adhesive strength value of the adhesive component was achieved at Q = 100 (g/mm), t = 160(^{0}C), U = 6 (m/min).
- In order to achieve the optimal adhesion strenght over the through feeding wrapping process of furniture elements, the most favorable conditions are achieved at combination of low feeding speed and high temperature of the adhesive.

ACKNOWLEDGEMENT

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EFFECT OF NANOPARTICLES ON THE WOOD-WATER RELATIONS

Miklós BAK
University of Sopron, Simonyi Károly Faculty of Engineering, Wood Science and Applied Arts
Bajcsy-Zsilinszky u. 4, 9400 Sopron, Hungary
Tel: 0036 518 187, Fax: 0036 518 647, E-mail: bak.miklos@uni-sopron.hu

Róbert NÉMETH
University of Sopron, Simonyi Károly Faculty of Engineering, Wood Science and Applied Arts
Bajcsy-Zsilinszky u. 4, 9400 Sopron, Hungary
Tel: 0036 518 152, Fax: 0036 518 647, E-mail: nemeth.robert@uni-sopron.hu

Ferenc MOLNÁR
University of Sopron, Simonyi Károly Faculty of Engineering, Wood Science and Applied Arts
Bajcsy-Zsilinszky u. 4, 9400 Sopron, Hungary
Tel: 0036 518 187, Fax: 0036 518 647, E-mail: ferimoln@gmail.com

Abstract

Results of an experimental research about the effect of different titanate nanoparticles on the wood-water relations are shown in this paper. Different wood species were used for the experiments. Treatment of wood with the nanoparticles was performed by impregnation method. Different concentrations of nanoparticles were used. Investigated properties after treatment were shrinking/swelling coefficient, equilibrium moisture content (EMC), water uptake and moisture permeability. Beside these, colour change (CIELab) and mechanical properties (compression strength and surface hardness) were investigated as well. Overall, we can state according to our investigations so far, that the impregnation with nanoparticles was successful. Shrinking and swelling properties decreased remarkably in case of all the four investigated wood species. As a side effect of the treatments, a slight colour change could be observed as well. No effect on the mechanical properties could be found as a result of the treatment.

Key words: *nanoparticles; shrinking/swelling; water uptake; EMC; colour change.*

INTRODUCTION

Wood is in contact with air humidity in all utilization fields. In many cases, wood elements are used as space border. Furthermore, during the processing wood undergoes a drying process. In these cases, it is important to know the sorption and diffusion properties of the wood to be able to understand the expected moisture transport processes in the wood during utilization. Diffusion properties of wood are strongly dependent on wood species and anatomical directions, but climatic conditions and sample size are also important factors (Jalaludin et al. 2010, Pfriem et al. 2010).

Moisture uptake in wood occurs through diffusion and capillary flow. Below the fibre saturation point moisture can transported as water vapour in the lumens, or as bound water in the cell walls with the diffusion. In this phase of moisture movement can be modelled successfully with a diffusion front moving through the wood, causing smooth moisture content gradients in the different cardinal directions (Droin-Josserand et al. 1988). However, in reality the moisture transport in wood is suspected to be more difficult (Absetz and Koponen 1997).

The characteristic of modified woods' sorption behaviour usually includes slower reaction to relative humidity changes than natural wood; therefore, the moisture uptake is lower and slower (Hill 2006). Changes in wood-water relations due to modification are also strongly influenced, however, by the wood species. The phenomenon of modification is even more complicated, as usually all of the treatment parameters have influence on the wood-water relations (WPG, concentrations, temperature, duration, etc.). The moisture uptake rate can therefore be different after various treatments.

The utilization of nanoparticles to improve the properties of wood is not widely investigated recently. On the other hand, a lot of promising results were achieved with the use of nanoparticles in relation to the mechanical, combustion, hydrophobic and some other properties of different polymers, papers or textiles (Wang et al. 2006; Csóka et al. 2007; Chen and Yan 2012; Nypelö et al. 2012; Jiang et al. 2011; Sun et al. 2007; Textor and Mahltig 2010). Recently there is only limited information available about the utilization of nanoparticles to improve the wood properties, but all results are positive. With the use of different nanoparticles the moisture uptake is reducible, UV-protection,

mechanical properties and durability is improvable (Rassam et al. 2012; Niemz et al. 2010; Yu et al. 2011; Mahltig et al. 2008). In some cases, fire resistance could be improved as well (Shabir Mahr et al. 2012). According to the careful examination of the results mentioned above, for the research received nanoparticles can be selected (different titanate nanotubes and nanowires, nanozinc, titan dioxide, montmorillonite and other nanoclays, etc.).

The novelty of the planned research is to investigate some nanoparticle, which effect on wood properties and the applicability on wood is not known until yet. Instead of surface treatments a full cross-section treatment is planned which could make the service life of wooden products longer. The utilization of wood contributes to the sustainable development. The technical properties of most of the European wood species are in many respects behind some competing materials, which are originating from sources that are disadvantageous in aspect of sustainability (endangered tropical wood species, plastics). An important objective is the expressive improvement of the properties of European wood species.

OBJECTIVE

The main goal of this investigation was to determine the influence of nanoparticle impregnation on the wood-water relations. This is relevant because it results in improvement of dimensional stability – but is also important during the utilization. During the service life of a product the surrounding climate is regularly changing, thus the EMC, and therefore the dimensions, are changing too. Short time exposure to extreme climates (either high or low relative humidity) will not result in pronounced dimensional changes if the moisture uptake is damped. The nanoparticle impregnation is a promising method to reduce the shrinking and swelling and therefore investigations are necessary to prove the effect of the treatment on the water-related properties of wood.

MATERIAL, METHOD, EQUIPMENT

Pine (*Pinus sylvestris*) and beech (*Fagus sylvatica*) wood was used for the tests. For the impregnation of wood, two types of aqueous emulsions were used, namely hydrophobic titanate-nanowire (HTNW) and hydrophobic titanate nanotube (HTNT). Both emulsions were used with two different concentrations, namely 1 wt% and 2 wt%. This resulted in four different treatments. The emulsions are proprietary formulas of NanoBakt Kft. (Budapest, Hungary). The nanowires were comprised of particles with dimensions of 50-100nm in diameter and 1-10µm in length, furthermore the nanotubes 5-8nm in diameter and 100-500nm in length. Their specific size distribution was not available.

The samples were weighed as a first step. The impregnation was carried out according to the full-cell process in a vacuum chamber at 20°C. The impregnation process involved an initial vacuum phase at 100mbar for 30min. The chamber was then pressurised at atmospheric pressure for 60min. The surfaces of the specimens were then gently rinsed with water to wash away residual material and conditioned at a temperature of 20°C and a relative humidity of 65% for 20 days. Consequently, the final weight was measured and the weight percent gain (WPG, %) for each specimen was calculated according to equation (1):

$$WPG = \frac{m_{imp} - m_{initial}}{m_{initial}} \times 100\ [\%] \quad (1)$$

where:
$m_{initial}$: weight of the sample before impregnation [g]
m_{imp}: weight of the sample after impregnation [g]

Colour Change

Colour measurements were carried out with a colorimeter (Konica-Minolta 2600d). The CIELab colour coordinates were calculated based on the D65 illuminant and 10° standard observer with a test-window diameter of 8 mm. The relatively large window was chosen to measure the average colour of earlywood and latewood regions combined. The radial surface of the sample was used for colour measurement. The colour of randomly chosen 3 points were measured on each sample. Measurements on samples were carried out before and after impregnation., and the total colour change (ΔE^*) was calculated.

Shrinking and swelling

Samples with the dimensions of 20×20×30mm (rad×tang×long) were used. 20 samples for each treatment, and 20 untreated samples served as control. After the impregnation, the samples were climatized at 20°C and 65% relative humidity until constant mass. After climatization samples

were dried at 103±2°C. Radial and tangential dimensions of the samples were measured before and after the drying. Also weighing of the samples was carried out before and after the drying. From these data the shrinking coefficient was calculated in both radial and tangential direction, according to equation (2):

$$SH_{coeff} = \frac{l_{wet} - l_{dry}}{l_{dry} \times U} \times 100 \quad (2)$$

where:
l_{wet}: radial or tangential dimension before drying [mm]
l_{dry}: radial or tangential dimension after drying [mm]
U: moisture content of the sample [%]

After drying, the same samples were immersed into water for 10 days. Radial and tangential dimensions of the samples were measured before and after the immersion. Also weighing of the samples was carried out before and after the immersion. From these data the swelling coefficient was calculated in both radial and tangential direction, according to equation (3):

$$SW_{coeff} = \frac{l_{wet} - l_{dry}}{l_{wet} \times U} \times 100 \quad (3)$$

where:
l_{wet}: radial or tangential dimension after immersion [mm]
l_{dry}: radial or tangential dimension before immersion [mm]
U: moisture content of the sample [%]

Water uptake

Samples with the dimensions of 10×50×50mm (rad or tangential×tang or radial×long) were used. 20 samples for each treatment, and 20 untreated samples served as control. Water uptake through both radial and tangential surface was measured. After the impregnation, the samples were climatized at 20°C and 65% relative humidity until constant mass. Samples were sealed at the edges and at one radial/tangential surface and weighed. Samples were then immersed to water with the unsealed surface and weighed at 2, 4, 8, 24, 48 and 72 hours. Water uptake was calculated according to equation (4):

$$W = \frac{m}{A} \quad \left[\frac{g}{m^2}\right] \quad (4)$$

where:
m: mass of the samples [g]
A: radial or tangential surface area of the samples [mm^2]

Equilibrium moisture content (EMC)

Samples with the dimensions of 20×20×30 mm (rad×tang×long) were used. 5 samples for each treatment, and 5 untreated samples served as control. Samples were dried at 103±2°C and weighed, then climatized at 20°C and 65% relative humidity until constant mass. After climatization, the samples were weighed again. EMC was calculated according to equation (5):

$$EMC = \frac{m_{wet} - m_{dry}}{m_{dry}} \times 100 \quad (5)$$

where:
m_{wet}: wet weight of the samples
m_{dry}: dry weight of the samples

RESULTS AND DISCUSSION

Retention

The amounts of chemical retention for tested specimens are shown in Fig. 1. There were no notable differences in chemical retention based on wood species, but significant differences based on nano-suspension concentration. The retention was increasing quite proportionately with the preservative concentration as the mean ratio of retention is 1,96 - 2,12, depending on wood species

and nanoparticles type. This result complied with the ratio of nano-suspension concentrations used during our experiments 2. It showed that the decay specimens effectively absorbed the nano-suspensions.

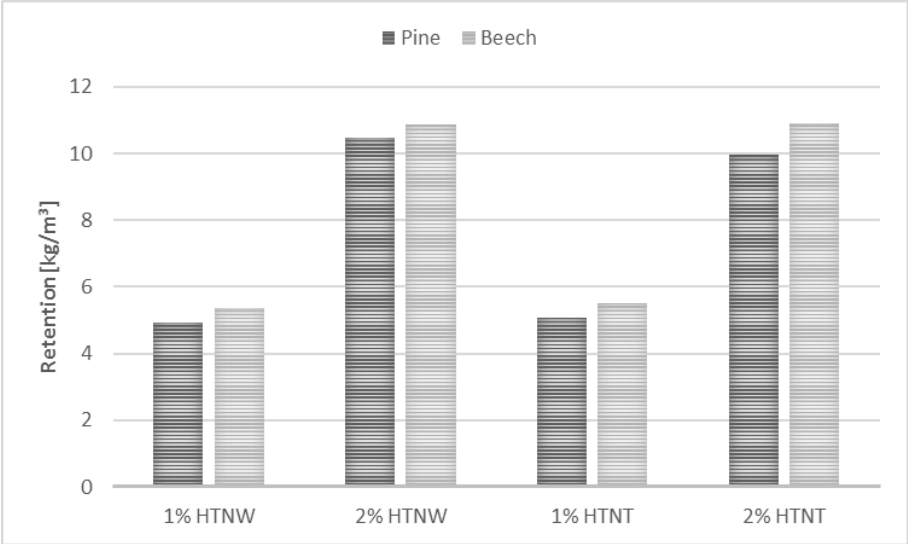

Fig. 1.
Retention of nanoparticles as a result of impregnation.

Colour Changes

A slight colour change could be observed as a result of nanoparticle impregnation. Total colour change values were in the range of 2,5 - 5,5, which is a region of slightly visible to well visible for the naked eye (Fig. 2.). However, only the treatment with 1% HTNW resulted in a well visible colour change. In case of pine no significant differences in colour change could be found between the different impregnations. In case of beech, the increasing concentration of the nanoparticles resulted in decreasing colour change. The colour change was visible as a fading (whitening) of the initial colour.

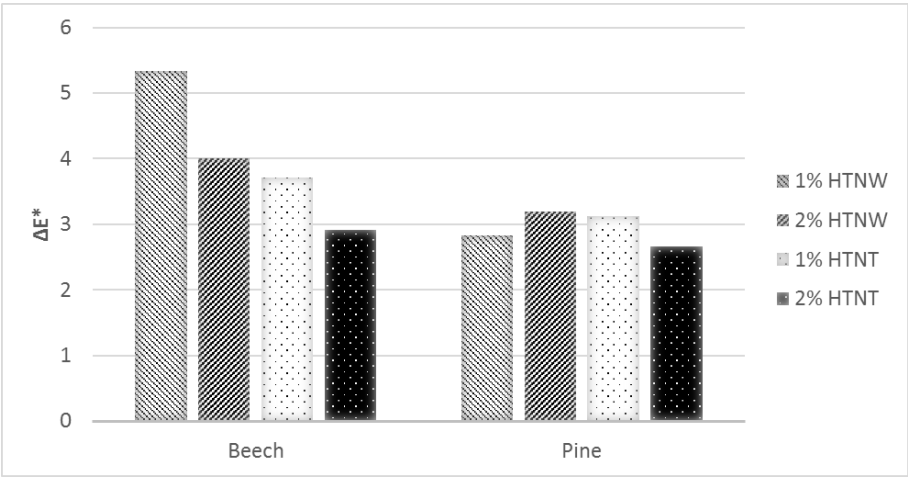

Fig. 2.
Colour change as a result of nanoparticle treatment.

Shrinking and swelling

Shrinking coefficient decreased in most cases as a result of nanoparticle impregnation. An interesting result is that the treatment decreased the shrinking coefficient more effective in radial direction, compared to the tangential direction (Fig. 3.). A possible reason for that can be a better penetration of the nanoparticles in radial direction, through the rays. No correlation could be found between the efficiency and the concentration of the nanoparticles in the suspension. However, the retention of the samples showed the same ratio than the ratio between the initial concentrations (~2). The used nanoparticles are relatively large in one dimension (length is 100-500nm for HTNT and 1-10µm for HTNW), so they have a stick-like shape. On the one hand, the dimensions of the particles

are probably too large for a good penetration into micro- and nanopores of the cell wall. On the other hand, the shape of the particles is not optimal for the penetration into the micro- and nanopores of the cell wall. This can lead to an uneven distribution of the nanoparticles in the wood material, and especially a weak penetration into the cell wall, which would be a key factor for a better efficiency.

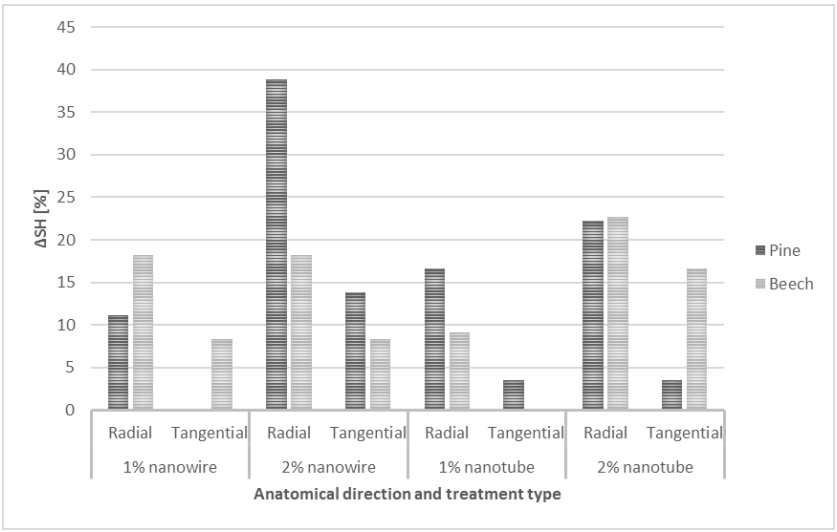

Fig. 3.
Decrease in the shrinking coefficient as a result of nanoparticle treatment.

After the shrinking test, the same samples were immersed to water to accomplish the swelling test. The efficiency of the treatment increased after this step in radial direction in case of the most treatments (Fig. 4.). In tangential direction, it remained unchanged or increased slightly as well, which might be a result of a leaching effect. This phenomenon is explained by the hydrophobic properties of the used nanoparticles. After the cell wall dried, the nanoparticles kept away the water more efficiently and this resulted in a lower swelling.

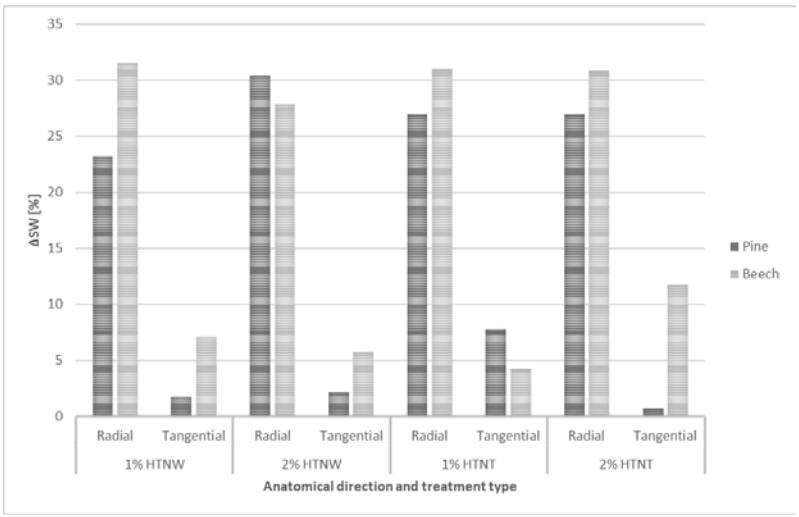

Fig. 4.
Decrease in the swelling coefficient as a result of nanoparticle treatment.

Water uptake

Water uptake decreased as a result of the nanoparticle treatments, where the effectiveness was better in case of tangential surface (water uptake in radial direction) (Fig. 5-6.). HTNT impregnation was more effective then impregnation with HTNW. The difference between the different concentrations was not significant. The hydrophobic property of the nanoparticles can keep away the water from the cell wall, but these results showed again the possible uneven distribution of the nanoparticles in the cell wall.

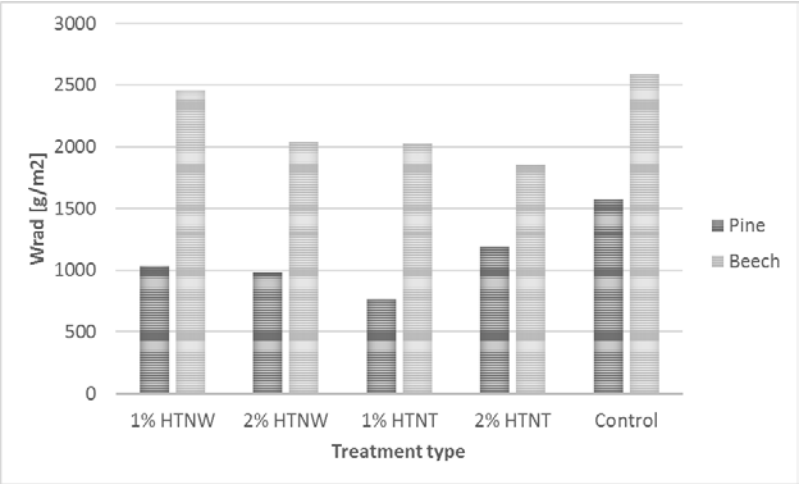

Fig. 5.
Water uptake during 72 hours immersion in water in radial direction.

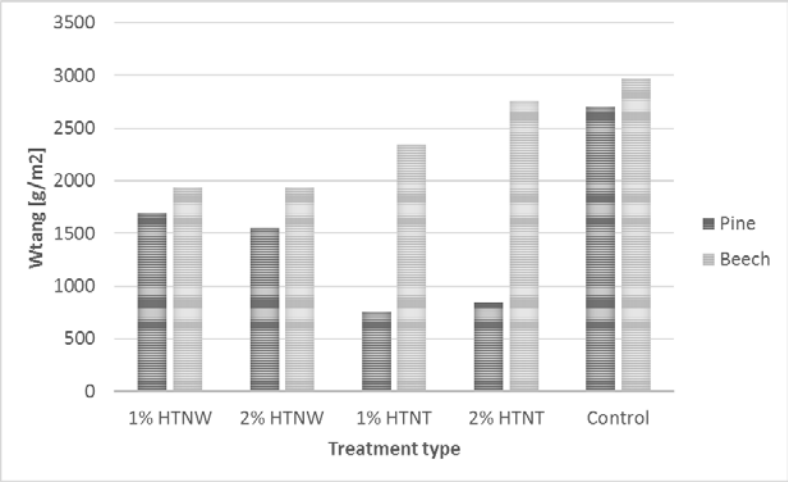

Fig. 6.
Water uptake during 72 hours immersion in water in tangential direction.

Equilibrium moisture content

There was no significant difference between the equilibrium moisture content of the treated and untreated samples. This result shows, that however the nanoparticles used are hydrophobic and can the liquid water keep away from the cell wall, the access of water vapour to it is not blocked.

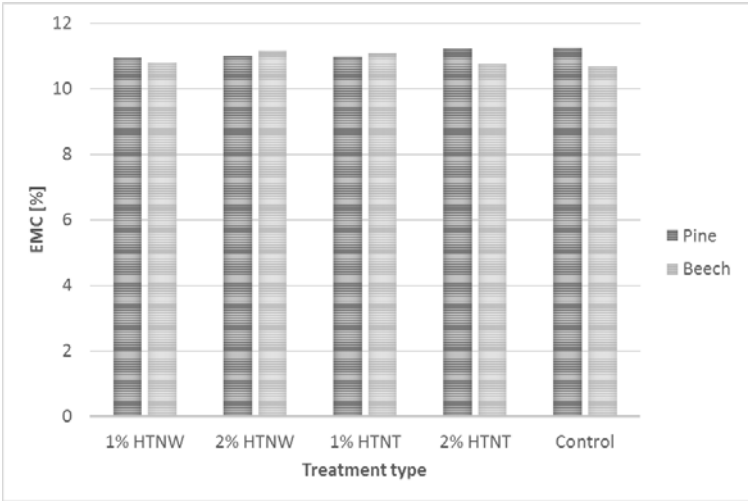

Fig. 7.
Equilibrium moisture content of the impregnated and control samples.

CONCLUSIONS

A slight colour change could be observed as a result of nanoparticle impregnation. The colour change was visible as a fading (whitening) of the initial colour. Shrinking and swelling could be decreased by the treatments, but there are differences in the effectiveness between the anatomical directions. A possible reason for that can be a better penetration of the nanoparticles in radial direction, through the rays. Furthermore, the distribution of the nanoparticles seems to be uneven in the cell walls, due to the unoptimal shape and length of the particles. Water uptake decreased as well and HTNT treatment was more effective compared to HTNW treatment. EMC remained unchanged after treatments.

The treatments gave an effective protection against shrinking and swelling, but the water uptake decreased only slightly in most cases and the EMC remained unchanged. Taking into consideration these results, we can state that the mode of action of the impregnation with HTNW and HTNT particles is a physical blocking of the penetration of liquid water into the cell wall, which is a physical bulking effect supplemented by the hydrophobic properties of the particles.

ACKNOWLEDGEMENT

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REVIEW OF POSSIBILITIES FOR ENHANCEMENT OF SURFACE PROPERTIES OF WOOD WITH HYDROTHERMALLY DEPOSITED TiO_2 PARTICLES

Pavel PORI
University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology, and Chemcolor Sevnica d.o.o.
Dolnje Brezovo 35, 8290 Sevnica, Slovenia
E-mail: pavli.pori@chemcolor.si

Marko PETRIČ
University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology
Jamnikarjeva 101, 1000 Ljubljana, Slovenia
Tel: 00386 1 3203620, E-mail: marko.petric@bf.uni-lj.si

Abstract

The novel low temperature hydrothermal process of TiO_2 particles deposition on wood was developed. The crystal structure of the hydrothermally made rutile particles, their morphology their distribution in wood and their interactions with lignocellulose were already reported in a paper in a scientific journal (Pori et al. 2016). However, some other results of investigations of the novel wood surface systems, consisting of hydrothermally deposited TiO_2 nanoparticles and the layers of transparent colourless coatings were presented on various conferences or have even not been published yet. Therefore, this paper brings an assembled review of our own results, in order to present the possibilities for enhancement of surface properties of wood in outdoor exposure with hydrothermally deposited TiO_2 particles.

It was established that the process of hydrothermal deposition of titania nanoparticles has both negative as well as positive effects when outdoor applications of Norway spruce wood are considered. For example, the results of artificial accelerated ageing of surface systems on the basis of natural oils or some surface coatings were quite promising. In the case of oil and alkyd based surface systems, application of the titania deposits on wood and of the TiO_2 nanoparticles in the coatings resulted in considerably low colour changes after artificial accelerated ageing.

Key words: *spruce; outdoor uses; hydrothermally deposited TiO_2 particles; hydrophobicity; bending strength; artificial accelerated ageing.*

INTRODUCTION

Due to its mechanical properties and aesthetic appeal, wood has a high application potential for its use in constructions, as an insulation material and for production of furniture (Hayoz *et al.* 2003). Unfortunately and particularly when outdoor applications of wood are regarded, some drawbacks of this sustainable material have to be considered. Because of its hydroxylated nature, wood shows poor dimensional and optical stability. In outdoor applications, the combination of UV light (Todaro *et al.* 2015) with moisture (Rassam *et al.* 2011) and temperature (Esteves *et al.* 2013), can lead to destruction of the lignocellulosic network, changes of wood's natural colour and to wood degradation (Deka *et al.* 2008). The most common approach to prevent wood from negative impacts of weathering is surface treatment of wooden elements with surface finishes. Most usually, the expected functions of a finish are both protective and aesthetic. Aesthetically, there is a growing trend among homeowners to maintain the natural look of the wood's original colour. But, the best protection from ultraviolet radiation is achieved by pigmented products, which cover the wood´s natural grain and texture (Daniel et al. 2004).

Colour of wood, when treated with a pigmented paint, becomes darker and the texture may lose its clearness by becoming hazed. A possible solution is incorporation of various nanoparticles into a coating (Sow et al. 2011, Fufa et al. 2012). Nanoparticles are small enough to provide excellent scattering and absorption of UV light, while coatings remain transparent with natural appearance of the wood (Bulian and Graystone 2009). To achieve better compatibility between nanoparticles and polymer matrix in a coating, their modification For instance, to improve dispersability of TiO_2 rutile nanoparticles in acrylic water based coating, two step surface modification of TiO_2 nanoparticles with Al_2O_3 and polyhedral oligomeric silsesquioxanes was investigated by Godnjavec et al. (2012). Another approach of application of nanoparticles is their deposition on surfaces to be protected. Rassam et al. (2012) reported the successful deposition of nano TiO_2 particles on wood, to improve the wood

stability against ultraviolet (UV) light and moisture degradation. The final step was exposure of wood samples to 120 °C - 150°C, that are very high temperatures for wood.

We developed a low temperature approach for the deposition of rutile TiO_2 particles on a Norway spruce wood surface by hydrolysis of $TiCl_4$ in aqueous solutions acidified with HCl, and crystallization at 75 °C and 90 °C (1 h) (Pori *et al.* 2016). In the mentioned paper, the focus was on studies of the crystal structure of the hydrothermally made rutile particles, their morphology, and their distribution in wood. It was shown that TiO_2-wood coordinative bonds of titanium ions with hemicellulose and lignin were established. TiO_2 deposited on wood treated with sodium dodecyl sulphate (SDS) became hydrophobic, contrasting the properties of untreated wood with a deposited TiO_2 particle coating, which remained hydrophilic (Pori *et al.* 2016).

OBJECTIVE

The mentioned paper of Pori and co-authors (Pori *et al.* 2016) reported the most important results of PhD dissertation of Pori (2016). Some other results from his doctoral dissertation were also already published, predominantly on various national and international conferences and some other results have not been published yet. So, the main aim of this paper, reviewing of our own results, is to report sensibly assembled results from previous years, in order to systematically present the possibilities for enhancement of surface properties of wood in outdoor exposure with hydrothermally deposited TiO_2 particles.

MATERIAL, METHOD, EQUIPMENT

For all tests, spruce wood (*Picea abies* (L.) Karst.) was used. The size of the samples was dependent on the test, as described in subsequent paragraphs

Hydrophobic properties and water-vapour transmission

For the purpose of determination of equilibrium moisture content (EMC) and water vapour transmission, the samples of the following size were prepared: 70 mm (radial) × 70 mm (axial) × 25 mm (tangential). Half of samples were modified with hydrothermal synthesis of TiO_2 on their surfaces, as described in details in our previous papers (Pori *et al.* 2012, Pori *et al.* 2016). After conditioning of the modified and non-modified samples, they were treated with the polyacrylic coating (in the following text named as "acrylic", Lesoton Aqua, produced by Chemcolor Sevnica, d.o.o., Slovenia) or with the organofunctionalised fluorinated silane surface system ("silane", Silles by Chemcolor). The acrylic paint was applied in two layers (2 x 100 g/m^2) and the application rate of the silane surface system coating was 180 g/m^2. The tests were performed also with the titania modified samples without coatings and with the control specimens.

Contact angles of water were determined by the sessile drop method, and the water vapour transmission and diffusion coefficient were determined by the standard method ISO 7783-1, by the Payne cup. More experimental details can be found in the paper of Pori and co-authors (2012).

Mechanical properties

Bending strength was determined with Zwick Z100 (6 specimens per one series), in accordance with the standard method (Pori 2016).

Resistance of various surface systems against accelerated artificial ageing

As a substrate, spruce wood samples without and with deposits of TiO_2 particles were used. The plates of the dimensions of 150 mm (L) × 50 mm (R) × 5 mm (T) were applied. In some cases the substrate's surfaces were pre-treated with sodium dodecyl sulphate, and deposition of TiO_2 particles (of the rutile or of the anatase crystalline form) was carried out from mixtures of aqueous solutions of $TiCl_4$ and HCl, where the concentration of HCl varied between 0 mol/L to 1 mol/L (Petrič *et al.* 2014). The following finishes were applied: a natural oil based finish, and coatings of a polyacrylate (OIL), polyalkyd (ALKYD) and a polyurethane (PU) types. Into the coatings, pre-treated (coated) nanoparticles of TiO_2 were dispersed, or the coatings without nanoparticles were applied. TiO_2 (mTiR) to be dispersed in the coatings was purchased from Cinkarna Celje d.d., Celje, Slovenia. As a surface modifier for the nanoparticles, the polyhedral oligomeric silsesquioxane trisilanol (POSS) was used. The process of surface coating of the nanoparticles is described in (Petrič *et al.* 2014). Into the coatings, the pre-treated (surface modified) TiO_2 nanoparticles were dispersed with the Ultra Turrax Ika T 25 for 15 min at 8000 min^{-1} – 10000 min^{-1}.

The finishes were applied to wood specimens manually, by brushing, and therefore the film thickness could not be controlled precisely. The coated samples were left to dry at ambient conditions for one day and on the next day, the second layer of a finish was applied, and on the third day, we

applied the third layer (only in the case of the oil based finish). In all cases, approximately total of 200 g/m^2 of the finish was applied.

Artificial accelerated ageing was carried out by exposure of the specimens in the Atlas SUNTEST® XXL Light Exposure and Weathering Testing Instrument, with three air-cooled 2100 W xenon lamps in a controlled chamber temperature, with an ultrasonic humidification system for humidity control and a specimen spray system. For the artificial accelerated weathering, the pre-programmed standard test in accordance with the EN ISO 11341 1A:2005 standard was applied. The influence of ageing was followed by colour and gloss measurements and by determination of contact angles of water.

Colour was measured in accordance with the standard ISO 7724-2 (1984) with the spectrophotometer SP62, X-Rite GmbH - OPTRONIK™, and gloss with the X-Rite AcuGloss TRI according to SIST EN ISO 2813 (60°). Finally, the sessile drop technique was used to determine the apparent contact angles of water on the specimens. This method is based on the observation of the profile of the drop deposited on the surface of the solid. The contact angle of water on each surface system was determined 1 second after deposition of a drop.

RESULTS AND DISCUSSION

Hydrophobic properties and water-vapour transmission of surface treated and non-treated wood, modified with TiO_2 deposits

To prolong durability of wood used outdoors, surface coatings for protection of wood against uptake of water vapour are normally applied. They should increase hydrophobicity of surfaces in order to protect wood against liquid water - rain. In our investigations with TiO_2 deposited wood and selected coatings (Pori ***et al.*** 2012) it was shown that the most hydrophobic surfaces were that of the silane treated ones, with the water contact angle of more than 133°. In average, the contact angles of water on the acrylic coated surfaces were 65.4°. Quite hydrophobic was also wood itself (90.7°). When we compare contact angles on surface coated non-modified wood with those on the coatings on the modified wood surfaces with TiO_2 deposits, we see that modification of wood has a slightly positive influence. The contact angles were in both cases slightly increased (acrylic 6.5°, silane 2.1°). On the contrary, deposition of TiO_2 on wood that was subsequently not treated with the polymer coating, caused more hydrophilic character of the samples. The decrease of contact angle (Figure 1) from 90.7° at the control specimens to 59.4° in average was observed.

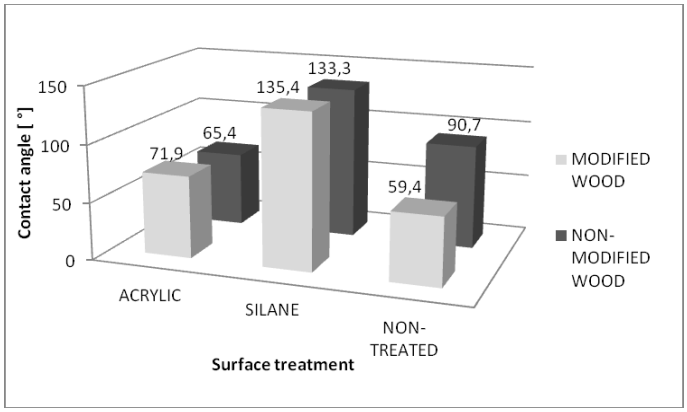

Fig. 1.
The contact angle of water on modified and non-modified wood, either treated or nontreated with acrylic or silane based coatings.

Due to modification (deposition of TiO_2 and also degradation by HCl), wood had a slightly increased equilibrium moisture content (EMC) (14.66% - 13.77%) compared the EMC of non-modified wood (12.77% - 12.59%) (Table 1). In general, the modified samples (no matter of the final surface treatment) achieved higher EMC compared to the non-modified ones. The non-modified samples (Table 1) exhibited differences in EMC due to orientation (UP or DOWN) of the treated surface in the cup only at acrylic coatings. The samples with the upside DOWN orientation of the treated surface had a higher average EMC. However, the specimens with TiO_2 deposits showed much higher EMC then non-modified ones (Table 1). Based on the analysis of the results from Table 1, we can assume that the bottleneck for internal mass transfer resistance of water is the surface system.

Water vapour diffusion coefficient and WVT through the systems had a similar character and

they were mostly related to the surface system of the sample. The samples with acrylic films had the lowest WVT and diffusion coefficient without any significant difference between modified and non-modified samples (Table 2). On the other hand, silane penetrated into wood cells, what was preliminary detected by optical microscope. Hydrophobicity was ensured due to chemical composition of silane and maybe also due to the nanostructure of the surface, but the net is open for the vapour transmission, just like in non-treated wood. However, the modified silane treated and non-treated samples had even larger WVT and diffusion coefficient values than the non-modified wood.

The fact that the synthesized TiO_2 exhibited hydrophilic influence on the surface is not favourable in terms of outdoor applications. Also, the same can be concluded for TiO_2 modified surface finished and non-finished wood - it unfortunately exhibited higher water vapour transmission and diffusion of the system.

Table 1

Equilibrium moisture content of samples, conditioned at air humidity of 65 % (u_E 65%) and at high relative air humidity; the samples were during the cup test with the treated surface facing up (u_E UP) or down (u_E DOWN)

	Modified* wood + acrylic coat	Modified wood + silane coat	Modified wood	Non-modified wood + acrylic coat	Non-modified wood + silane coat	Control
$u_{E\ 65\%}$ [%]	14.66	13.79	13.77	12.59	12.77	12.69
$u_{E\ UP}$ [%]	21.60	30.19	29.27	16.88	22.92	22.77
$u_{E\ DOWN}$ [%]	43.69	27.45	26.55	27.09	22.93	23.05

* modified means wood with titania deposits

Table 2

WVT and diffusion coefficient of modified and non-modified surface treated and nontreated samples, depending on orientation of the sample in the Payne cup

	Modified* wood + acrylic coat	Modified wood + silane coat	Modified wood	Non-modified wood + acrylic coat	Non-modified wood + silane coat	Control
WVT_{UP} [g/^{2} 24h]	1190	3545	3147	1188	2086	2257
WVT_{DOWN} [g/^{2} 24h]	1019	2740	2692	1166	2191	2374
D_{UP} [m^3/m s]	8.50×10^{-13}	2.75×10^{-12}	2.25×10^{-12}	8.19×10^{-13}	1.43×10^{-12}	1.63×10^{-12}
D_{DOWN} [m^3/m s]	7.64×10^{-13}	2.17×10^{-12}	1.92×10^{-12}	8.27×10^{-13}	1.52×10^{-12}	1.73×10^{-12}

* modified means wood with titania deposits

Bending strength and modulus of elasticity of wood with titania deposits

Although the bending strength and modulus of elasticity are not surface properties, we included these properties in this review, since they are important parameters when wood is used for construction elements, what can be a common situation when it is used in outdoor applications. It turned out that because of exposure of wood to the process of titania deposition, mechanical properties decreased (Table 3). As can be seen, the bending strength was dependent on the deposition reaction time. After 15 min of treatment the bending strength decreased for 13.5 %, and after 30-minutes for 16.8%.

Table 3

Bending strength (F_m) and modulus of elasticity (E_L) of the treated and untreated samples

Samples:	Control	15 min titania deposition process	30 min titania deposition process
F_m [N/mm^2]	98.4	85.1	81.9
Std.dev	15.0	11.9	12.4
E_L [N/mm^2]	13000	11400	11700
3 months	2300	1390	1830

Influence of artificial accelerated ageing on colour, gloss and hydrophobicity (Petrič et al. *2014*)
As can be seen from Table 4, application of surface coating systems on the substrates, either without or with the TiO_2 deposits, quite substantially changed the appearance of wood, in terms of the ΔE colour difference value. In Table 5 there are presented the ΔE values of the coated specimens after 100, 200, 300, 400 and 500 hours of artificial accelerated ageing. From the data in Table 5 it can be seen that the specimens coated with the oil based finish, alkyd type coating or with the 2-K polyurethane coating, without TiO_2 deposits and without TiO_2 nanoparticles in the coating systems, even after only 500 hours of artificial ageing changed their colours quite substantially. From ΔE = 13.2 for the oil and alkyd based finishes to ΔE = 14.4 for the polyurethane type coating. On the other hand, the influence of TiO_2 deposits on the substrate and of TiO_2 particles in the coating formulation was obviously dependent on the coating system. In the case of the polyurethane, we could not observe any substantial improvement of the colour stability. On the other hand, TiO_2 deposits on wood substantially decreased coloured changes of oiled specimens (ΔE = 5.3 and 4.6 at surfaces with deposits and oil or with deposits and oil with TiO_2, respectively). However, when the oil with TiO_2 particles was applied to the substrate, covered with TiO_2 deposits, the colour change was surprisingly even larger than in the case of the oil based finish without TiO_2 on the substrate without the deposits. This can lead to the conclusion that the TiO_2 deposits on the substrate are essential for colour stabilisation and not the titania particles in the oil. Somewhat different behaviour was observed at the alkyd type coating. The best result in terms of the colour stability during ageing was observed at the alkyd with nanoparticles, but applied to the non-modified substrate (ΔE = 4.5), followed by the formulation with nanoparticles, applied on the surface with titania deposits (ΔE = 5.6). This observation anticipates a kind of a synergistic activity of both the TiO_2 particles on wood as well as in the coating formulation.

Table 4

Changes of colours due to application of surface finishes and the values of L*, a* and b* of the coated samples prior to exposure to artificial accelerated ageing

The system	ΔE due to application of the coating	Colour of the coated sample		
		L*	a*	b*
OIL	5.54	75.08	8.33	38.16
Deposits + OIL	13.59	57.11	16.89	33.90
OIL(TiO_2)	11.46	65.58	14.00	39.43
Deposits + OIL(TiO_2)	7.13	62.61	15.04	30.81
ALKYD	3.81	74.94	8.49	38.18
Deposits + ALKYD	19.05	68.15	10.20	31.11
ALKYD(TiO_2)	13.83	76.28	7.97	20.95
Deposits + ALKYD(TiO_2)	6.51	71.73	8.26	15.94
PU	1.82	74.85	8.38	34.21
Deposits + PU	5.81	61.51	13.69	28.81
PU(TiO_2)	17.69	77.32	7.61	16.27
Deposits +PU(TiO_2)	17.76	70.69	9.02	11.38

Table 5

ΔE values in dependence of artificial accelerated ageing

The system	ΔE after accelerated ageing				
	100 hours	200 hours	300 hours	400 hours	500 hours
OIL	6.56	9.03	8.24	7.99	13.25
deposits + OIL	1.75	3.67	2.22	2.79	5.28
OIL(TiO2)	5.94	9.09	13.89	16.53	22.43
deposits + OIL(TiO2)	1.09	1.77	2.10	2.46	4.57
ALKYD	7.04	9.48	10.08	10.49	13.19
deposits + ALKYD	6.26	9.55	10.28	10.87	14.14
ALKYD(TiO2)	3.99	4.20	5.07	5.03	4.47
deposits + ALKYD(TiO2)	2.22	3.01	3.76	4.65	5.58
PU	8.13	12.22	14.59	14.18	14.44
deposits + PU	6.99	11.11	12.09	12.45	14.19
PU(TiO2)	7.31	10.65	11.01	11.81	13.76
deposits + PU(TiO2)	7.21	10.00	10.50	11.00	12.40

* The best results are emphasised by grey background of the cells in the table

A decrease of gloss of the surface systems was also observed after artificial accelerated ageing of the oil and alkyd based surface systems. The polyurethane coating exhibited a relatively low gloss already before the exposure and any substantial influence of ageing on gloss was not observed in this case.

Similarly to the influence of gloss, artificial accelerated ageing caused also the decrease of the contact angle of water (Table 6). From the data in Table 6 it is hard to draw some general conclusions on the influence of ageing on the contact angle of water. Also, some firm correlations with the changes of colour and gloss cannot be established. Nevertheless, it is possible to conclude that contact angles on the oiled surfaces were the least affected by ageing and those on the surfaces with the polyurethane, the most.

Table 6

Contact angle in dependence of artificial accelerated ageing*

The system	Contact angle of water (°)					
	Before exposure	100 hours	200 hours	300 hours	400 hours	500 hours
OIL	73.6	83.1	74.0	72.0	70.5	72.3
deposits + OIL	78.4	82.6	78.3	76.3	74.3	77.1
OIL(TiO2)	103.1	101.5	74.9	73.3	64.5	46.2
deposits + OIL(TiO2)	79.2	84.2	72.2	73.1	69.0	68.9
ALKYD	85.2	77.2	60.2	57.0	47.1	50.5
deposits + ALKYD	80.0	71.0	59.1	56.6	46.6	50.5
ALKYD(TiO2)	78.2	90.8	77.9	86.2	55.8	56.3
deposits + ALKYD(TiO2)	74.1	76.5	68.0	52.2	41.8	51.0
PU	75.3	64.8	23.1	50.2	44.6	45.2
deposits + PU	74.4	64.2	49.9	29.4	45.6	34.9
PU(TiO2)	77.2	67.6	45.9	38.8	25.4	41.5
deposits + PU(TiO2)	83.8	68.5	49.8	38.8	29.4	30.4

* The best results are emphasised by grey background of the cells in the table

CONCLUSIONS

At TiO_2 modified samples, finished with acrylic or silane surface systems, the contact angle slightly increased in comparison with the contact angle on the samples without TiO_2 deposits. The equilibrium moisture content of the samples increased due to modification in the $TiCl_4$ / HCl solution. Also, both titania modified surface finished and non-coated wood exhibited increase in water vapour transmission and diffusion of the system. The process of titania particles depositition substantially decreased bending strength of the samples: after 15 min of treatment the bending strength decreased for 13.5 %, and after 30-minutes even for 16.8 %. The results of artificial accelerated ageing of some surface systems on the basis of oil, alkyd and polyurethane type coats were quite promising. In the case of oil and alkyd based surface systems, application of the titania deposits on wood and of the TiO_2 nanoparticles in the coatings resulted in considerably low colour changes after 500 hours of ageing. On the other hand, at the polyurethane coating, a positive influence of the deposits and nanoparticles was not observed.

In general, it seems that the low temperature hydrothermal TiO_2 deposition process is an interesting alternative to conventional protection systems for wood in outdoor application. It has so negative as well as positive influence on surface properties of treated wood, either not finished with a surface coating or with a surface finish. We believe, that this promising technique for surface treatment of wood should be extensively investigated in future.

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THE INFLUENCE OF ACETYLATION ON THE COLOUR AND PHOTOSTABILITY OF COMMON HORNBEAM WOOD (*Carpinus betulus* L.)

Fanni FODOR
University of Sopron, Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts
Bajcsy-Zs. Str. 4, 9400 Sopron, Hungary
Tel: 0036 99 518 399, Fax: 0036 518 647, E-mail: fodor.fanni@phd.uni-sopron.hu

Chiel LANKVELD
Accsys Technologies PLC Group
Westervoortsedijk 73, 6827 AV Arnhem, Netherlands
E-mail: chiel.Lankveld@accsysplc.com

Róbert NÉMETH
University of Sopron, Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts
Bajcsy-Zs. Str. 4, 9400 Sopron, Hungary
E-mail: nemeth.robert@uni-sopron.hu

Abstract

The aim of our research was to study the effect of acetylation and boiled linseed oil-coating on the photodegradation of common hornbeam wood (Carpinus betulus L.). For this study, a 200-hour-long mercury vapour lamp irradiation test was carried out. The tests were done on 4 different types of wood materials: native hornbeam, acetylated hornbeam, boiled linseed oil-coated hornbeam and boiled linseed oil-coated, acetylated hornbeam samples.

According to the results, the change of colour coordinates and the colour difference compared to the original colour was determined as well as conclusions were drawn on the photodegradation process according to the Fourier Transform Infrared spectra.

Key words: *hornbeam; acetylation; photodegradation; colour; FTIR.*

INTRODUCTION

The aesthetic appearance of wooden products are greatly influenced by their colour. This is why their colour stability is expected by users whether it is an indoor furniture or an outdoor decking. The colour of wood changes if it is exposed to ultraviolet (UV) light or thermal impact. The colour can be objectively determined with various methods. In this work CIELAB colour system was used where L* defines lightness (0 is black and 100 is white), a* denotes red/green hue (positive values for red and negative values for green), and b* denotes yellow/blue hue (positive values for yellow and negative for blue). The colour change of wooden products can be determined by calculating the change in colour components (ΔL, Δa, Δb) and then the total colour difference (ΔE*). According to Terziev and Boutelje (1998), and Mononen ***et al.*** (2002) the difference can be seen by the naked eye if ΔE* is 2 or more. Jirouš and Ljuljka (1999) determined the levels of colour differences for paper but these can be used for wood also (Straže and Gorišek 2008). It should be noted that the difference between wooden surfaces cannot be noticed just because of the colour change but also of the inhomogeneity of wood itself (vessels, tyloses, ray flecks, grain structure, early and latewood transition).

If the wood is exposed to natural weather, the colour change is affected by the temperature, sunny hours, precipitation, rate of UV-A and UV-B radiation, etc. The surface starts to grey after months because of the leaching of lignin, thus a* and b* decrease significantly (Tolvaj and Papp 1999, Tolvaj and Mitsui 2005).

The advantage of artificial ageing is the reproducibility of the measurements, the constant settings and the short testing time. In these test, artificial lightsources are used like xenon lamp, mercury-vapour lamp, etc. Only photodegradation occurs unlike in case of weather exposure.

The colour of objects is determined by the conjugated double bonds in their chemical structure. These bonds are present in lignin and extractives. The colour of wood is mainly defined by the quantity and quality of extractives. The changes in the chemical structure can be examined by Fourier Transform Infrared (FTIR) spectroscopy. Here the reflectance spectra can be converted to absorbance spectra with Kubelka-Munk (K-M) theory. The changes are better evaluated on differential spectra as the bands are clearly visible (Tolvaj 2013).

In the past decades, many researchers reported in the topic of photodegradation using mercury-vapour lamp (Ohkoshi 2002, Colom *et al.* 2003, Tolvaj *et al.* 2011, Tolvaj 2013). After UV exposure, the reduction in absorption of guaiacyl lignin (1510 cm-1) and syringyl lignin (1600 cm-1) indicates the degradation of lignin aromatic ring. The absorption of conjugated carbonyl groups initially decreases then increases. At 1765 and 1708 cm-1 band the absorption of unconjugated carbonyl groups increase.

Acetylation is a chemical wood modification method, which improves the durability, dimensional stability and strength of wood without being toxic to the environment (Hill 2006). In the process, the wood is impregnated with a liquid reagent using vacuum and pressure, so that it becomes integrated in the wood modifying its chemical structure and properties. On industrial level, acetic anhydride is used (Accsys Technologies, the Netherlands). When acetic anhydride reacts with the hydroxyl (OH) groups in the cell wall, acetyl groups form. These are bigger molecules than OH groups which results in a denser and heavier wood material. The properties of acetylated wood is generally given according to its WPG (Weight Percentage Gain) as the physical and mechanical properties usually improve by increasing the WPG.

The colour of wood does not necessarily change significantly after acetylation unlike in case of thermal modification, as it is done at lower temperatures. Broadleaved species usually darken at a higher rate than coniferous species, and dark-coloured species usually become brighter while light-coloured species usually darken (Rowell 2013, Mitsui 2010, Fodor 2015, Dong et al 2016).

After acetylation, the absorption of carbonyl groups (1740 cm-1) and methine (CH), methylene (CH2), methyl (CH3) groups (2970 cm-1) increase while the absorption of the functional groups of lignin decrease (Mohebby and Radjihassani 2008, Fodor *et al.* 2017).

In this work mercury-vapour lamp was used for the irradiation of wood. It results in a stronger colour change in a shorter time than xenon lamp or natural sunlight. This is because the mercury-vapour lamp has different wavelength emission. Unlike xenon lamp, it emits light in all UV regions. 80% of its emission is UV light, from which 31% is UV-A (380-315 nm) region, 24% is UV-B (315-280 nm) region and 25% is UV-C (> 280 nm) region. It cannot imitate the irradiation of natural sunlight but the progress of photodegradation can be observed in a short time (Tolvaj and Persze 2011).

OBJECTIVE

The aim was to examine the effect of acetylation on the photostability of hornbeam wood. The samples were to artificial irradiation (mercury-vapour lamp). The rate of photodegradation was evaluated according to the change of colour components and FTIR differential spectra.

MATERIAL, METHOD, EQUIPMENT

Edged and air-dry hornbeam boards were ordered from a Hungarian sawmill (BOPAÁR Ltd.). The dimensions were 27 × 160 × 2500mm (thickness × width × length). Half of the boards were left untreated and the other half was sent to Accsys Technologies to be acetylated under industrial conditions. The average WPG was 15%.

For the tests, half of the samples were coated with boiled linseed oil as it is of reasonable price, does not hide the grain pattern of wood and is of natural origin. It was layered two times.

The artificial ageing was carried out in a Sapratin ageing machine, at the Institute of Physics and Electronics at the University of Sopron. There were two mercury-vapour lamps used (800 Watt) which were 64 cm above the samples. The temperature of the equipment was set to 50°C.

The samples were of 20 × 45 × 140mm (thickness × width × length) with planed, smooth, tangential surface. There were five circles marked on each sample for colour measurement.

There were 10 hornbeam (marked H), 10 acetylated hornbeam (A), 10 boiled linseed oil-coated hornbeam and 10 boiled linseed oil-coated and acetylated hornbeam samples.

The colour was expressed in CIE L*a*b* colour space with X-Rite SP60 Portable Colorimeter and Color iControl program. The colorimeter's sensor head was 8mm. The colour was measured and calculated based on the D65 illuminant and 10° standard observer.

The samples for FTIR spectroscopy were of 5 × 10 × 30mm (thickness × width × length) with planed, smooth, radial surface. For the measurements JASCO FT/IR-6300 spectrometer and Spectra Manager program was used. The final spectra of each sample was the average of 45 measurements.

The colour and FTIR measurement was determined after 0-5-10-20-30-60-120-200 hours of irradiation.

RESULTS AND DISCUSSION

As a result of acetylation the colour of hornbeam became darker, greyish brown. The ray flecks became darker, the wavy grain has become more prominent to the naked eye. The modification process affected the whole cross section. The outer layer of wood (2-3 mm) is darker but it disappears after further processing.

After 5 hours of mercury-vapour lamp irradiation, the colour changed remarkably, especially in case of boiled linseed oil-coated acetylated hornbeam. The rate of colour change decreased over time (Fig. 1.).

During irradiation, hornbeam's light colour became darker yellow. The brightness decreased while the red and yellow hue increased (Table 1 and Fig. 1.). Fig. 2. shows the shifting of the yellow and red hue. The colour of hornbeam coated with boiled linseed oil changed similarly, the brightness decreased, the red hue increased, while the yellow hue initially (in the first 5 hours) decreased then increased. Coating hornbeam wood with boiled linseed oil improved its colour stability. (Fig. 3.).

Acetylated hornbeam's dark greyish brown colour brightened heavily as a result of mercury lamp irradiation. The biggest colour change was measured in the first 5 hours. The brightness increased, the red hue decreased, the yellow hue initially decreased then increased (Table 1 and Fig. 1.). The biggest total colour change was in case of boiled linseed oil-coated, acetylated hornbeam. (Fig. 1.). The change of its colour components were similar to acetylated hornbeam but the shifting of red and yellow hue was more prominent (Fig. 2.).

Table 1

Colour coordinates of hornbeam samples before and after exposed to mercury-vapour lamp for 200 hours

	Before irradiation			After irradiation (200 hour)		
	L*	a*	b*	L*	a*	b*
Hornbeam	80,71	2,93	18,92	72,64	7,77	33,10
Acetylated hornbeam	52,97	6,61	20,59	67,99	3,40	18,23
Boiled linseed oil-coated hornbeam	72,17	6,39	34,94	64,69	11,17	38,24
Boiled linseed oil-coated acetylated hornbeam	28,19	15,44	41,55	55,65	8,18	27,05

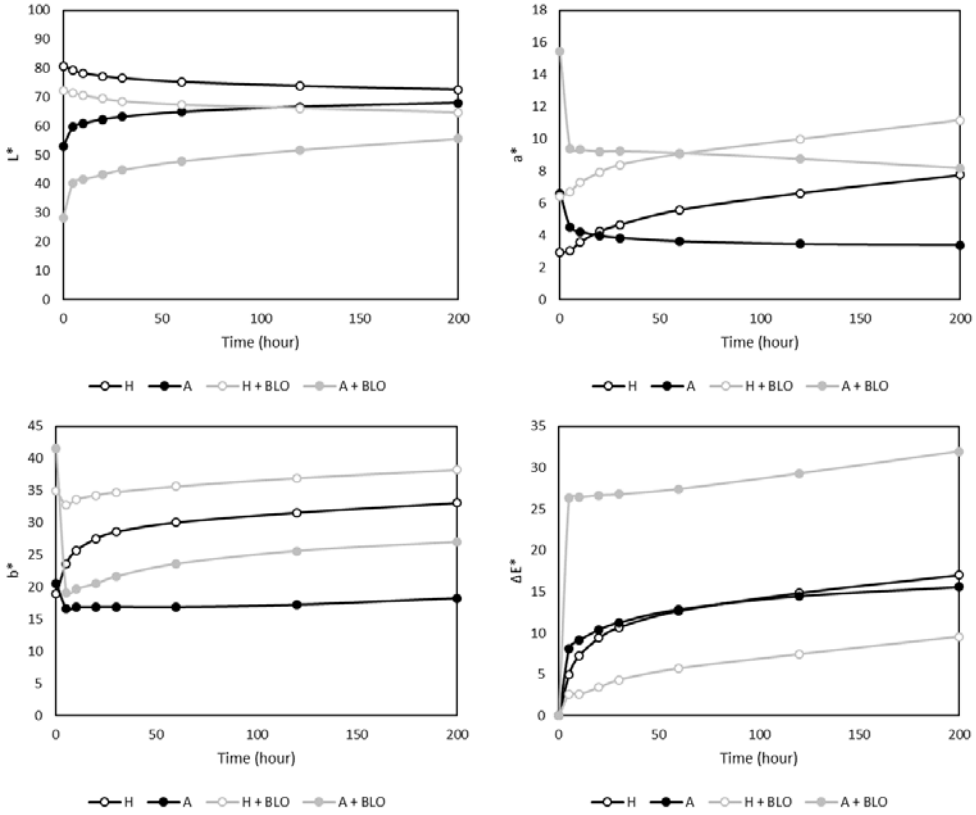

Fig. 1.

Change of colour coordinates during mercury-vapour lamp irradiation (L*: lightness, a*: red hue, b*: yellow hue, ΔE*: colour difference of the actual and the original colour, H: hornbeam, A: acetylated hornbeam, untreated or BLO: coated with boiled linseed oil).

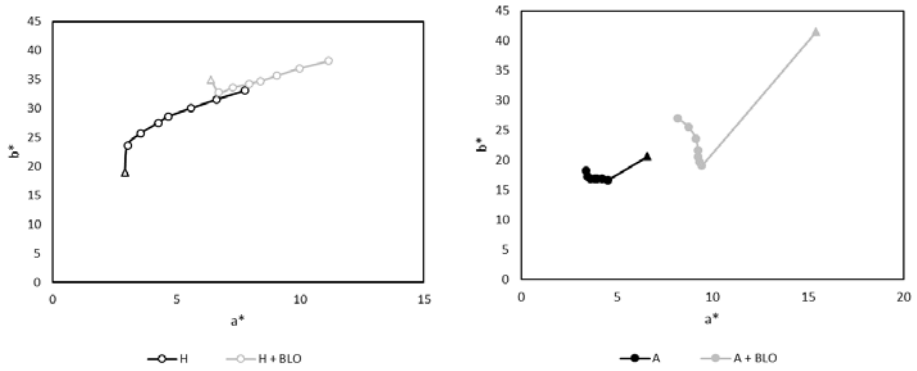

Fig. 2.
Change of red (a*) and yellow (b*) colour points during mercury-vapour lamp irradiation. The points marked with triangle represent the colour points of the original sample, then it is followed by the colours measured in the 5th, 10th, etc. hours (H: hornbeam, A: acetylated hornbeam, untreated or BLO: coated with boiled linseed oil).

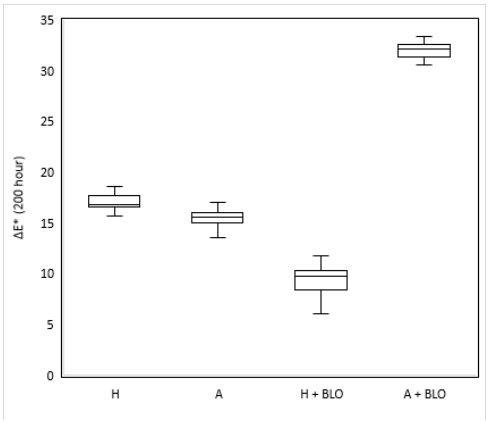

Fig. 3.
Box diagram of the colour changes (ΔE*) caused by 200 hour-long mercury-vapour lamp irradiation compared to the original colour (H: hornbeam, A: acetylated hornbeam, untreated or BLO: coated with boiled linseed oil).

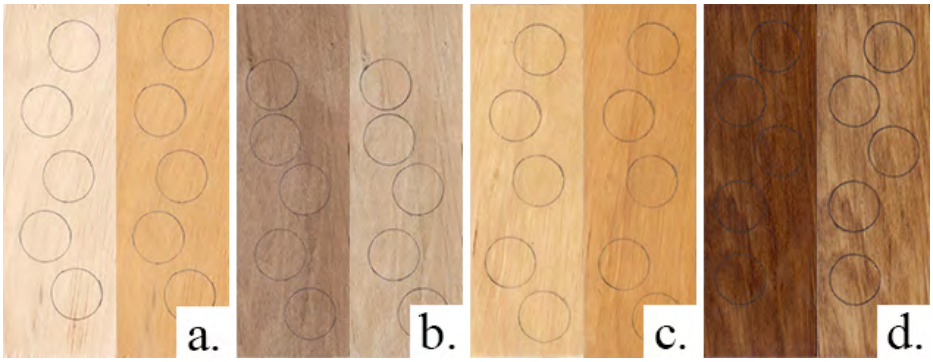

Fig. 4.
Photos of hornbeam samples before and after being exposed to 200 hour-long mercury-vapour lamp irradiation (a.: untreated hornbeam, b.: acetylated hornbeam, c.: boiled linseed oil-coated hornbeam, d.: boiled linseed oil-coated, acetylated hornbeam).

Fig. 4. shows the scans of one sample from each type before and after 200 hour-long irradiation. The yellowing of hornbeam and brightening of acetylated hornbeam is visible to the naked eye.

The FTIR spectra on Fig. 6. was measured before irradiation. After each phase (5-10-20-30-60-120-200 hours) the FTIR spectra was measured again. The changes were determined according to differential spectra, which was calculated by subtracting the initial (non-irradiated) spectra from the

irradiated spectra (Fig. 5.). In each spectrum the peaks and absorption bands were determined (Table 2) and marked with numbers on each diagram and in the text.

According to previous studies (Fodor 2015), the moisture content of hornbeam is greatly reduced due to the bulking of the cell wall during acetylation. As the cell wall's OH groups were replaced by acetyl groups, the weight increased by 15% (WPG). During UV irradiation the hydrogen bonds were broken, the OH groups changed and rearranged in the system which is indicated by the positive and negative peaks in the spectra (1). There are bigger peaks (differences) in the differential spectrum of acetylated hornbeam than untreated hornbeam.

The absorption of methyne (CH), methylene (CH_2) and methane (CH_3) groups increased after UV-B and UV-C irradiation except for acetylated hornbeam. This band is usually not affected by photodegradation. The reason for this absorption change can be due to the fact that this band cannot be separated from the band of OH stretching (Tolvaj and Faix 1995). Acetylated hornbeam has a bigger proportion of these groups than untreated hornbeam.

Cellulose is a rigid chain, linear polymer which is mostly crystalline in wood with some amorphous parts. The content and structure of cellulose is directly related to wood strength properties (Winandy and Rowell 1984). A previous research proved that there was no significant degradation in the cellulose of hornbeam after acetylation (Fodor 2015). The spectra show reduction in symmetrical C-H deformation (8), asymmetric C-O-C stretching (12), and symmetric C-O-C stretching (13), but increase in C-H deformation (9) and C-O stretching (14). Cellulose is more resistant to photodegradation, here the absorption reduction is associated with the change in hemicellulose.

Hemicelluloses have shorter polymer chain length as compared to cellulose, not crystalline and are composed of both hexose and pentose sugars. They are very reactive and they have proportionally the most hydroxyl groups (Winandy and Rowell 1984). This means when the OH groups were substituted by acetyl groups, the hemicellulose was mostly acetylated which increased the weight of the polymer. The absorption of conjugated carbonyl groups decreased slightly in acetylated wood, probably due to minor degradation of xylans in acidic medium (Fodor ***et al.*** 2017). As a result of UV irradiation, the aromatic rings of lignin rupture, carboxyl groups and/or lactones form, thus the absorption of carbonyl groups increase (Tolvaj and Faix 1995). The unconjugated carbonyl region has two distinct wavenumber ranges at 1800-1760 cm^{-1} and 1740-1700 cm^{-1} (3). In case of acetylated hornbeam the absorption at 1743 cm^{-1} is higher than at 1793 cm^{-1}. The absorption of hornbeam is smaller at 1698 cm^{-1} than at 1773 cm^{-1}. In every case, the absorption of conjugated carbonyl groups decreased (4). These peaks are less prominent in case of boiled linseed oil-coated samples. After acetylation the amount of carbonyl groups increased, thus the rate of photodegradation was higher. In case of non-acetylated samples, the thermally unstable acetyl groups degraded which indicates the reduction of carbonyl groups. There are positive and negative peaks as well in C-H deformation (8), C-O stretching (11), asymmetric C-O-C stretching (12) and symmetric C-O-C stretching (13) in hemicellulose, this can be due to the rupture of etheric bonds and reformation in the system.

Table 2
Wavenumber characterization of the infrared spectra of hornbeam samples according to Tolvaj (2013)

Band number	Wavenumber (1/cm)	Functional group	Assignment
1	3677-3152	Hydroxyl group (OH) stretching	Cellulose, hemicellulose, lignin
2	2983-2844	CH stretching	Methine (CH), methylene (CH2), methyl (CH3) groups
3	1804-1773 1745-1698	Unconjugated C=O (carbonyl group) stretching	Xylan
4	1675-1658	Conjugated C=O (carbonyl group) stretching	Xylan
5	1604-1596	Aromatic skeletal vibration	Syringyl lignin
6	1514-1495	Aromatic skeletal vibration	Guaiacyl lignin
7	1476-1411	Asymmetric C-H deformation	Lignin, carbohydrates
8	1397-1383	Symmetric C-H deformation	Cellulose and hemicellulose
9	1372-1309	C-H deformation	Cellulose
		C-OH vibration	Syringyl derivatives
10	1279-1266	Ring vibration	Guaiacyl lignin
11	1219-1215	C-O stretch	Xylan
12	1176-1159	Asymmetric C-O-C stretching	Cellulose and hemicellulose
13	1146-1117	Symmetric C-O-C stretching	Cellulose and hemicellulose
		Aromatic C-H skeletal vibration	Lignin
14	1102-1067	C-O vibration	Cellulose and hemicellulose

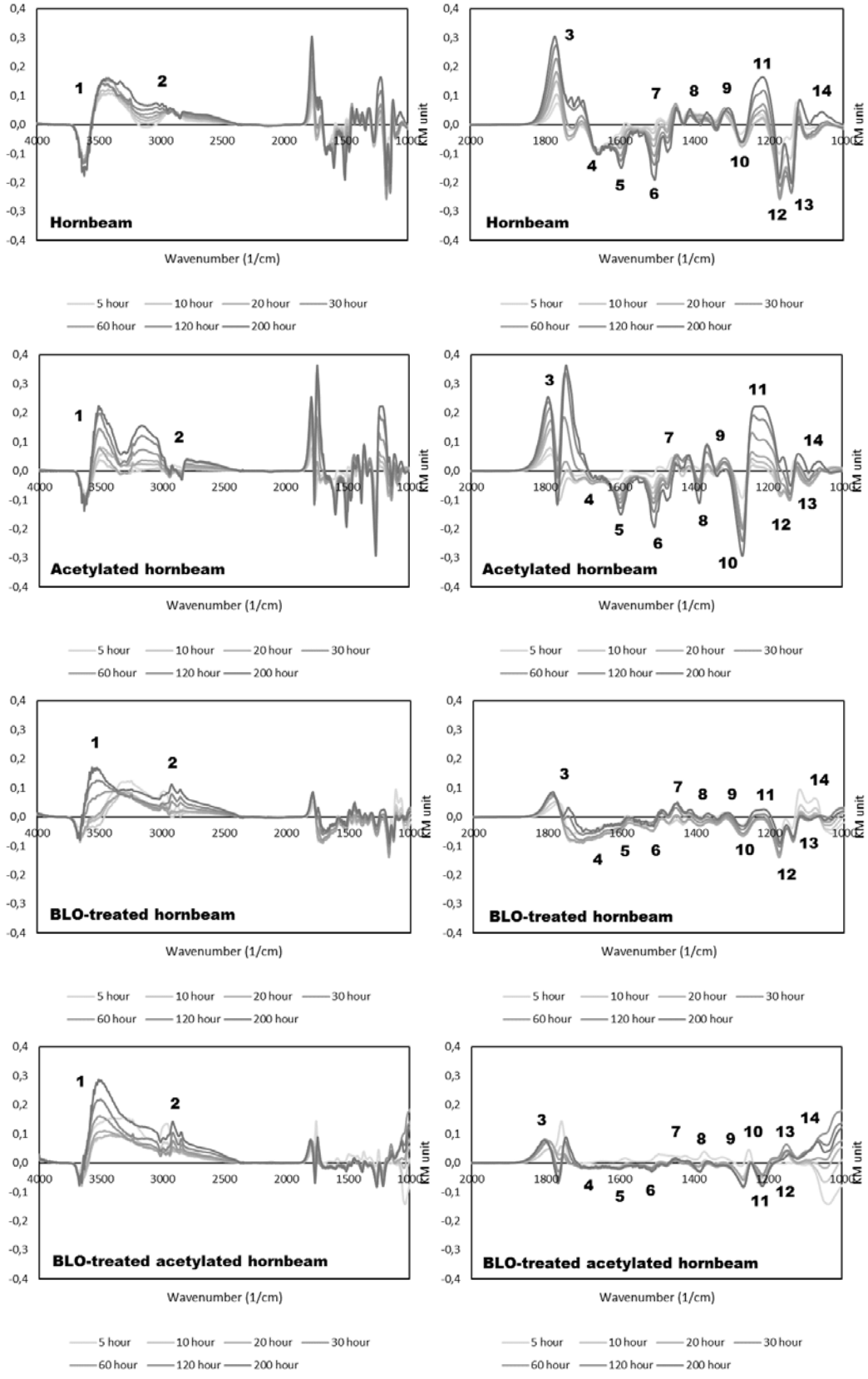

Fig. 5.
Change of hornbeam samples' FTIR difference spectra during 200 hour-long mercury-vapour lamp irradiation (BLO: boiled linseed-oil).

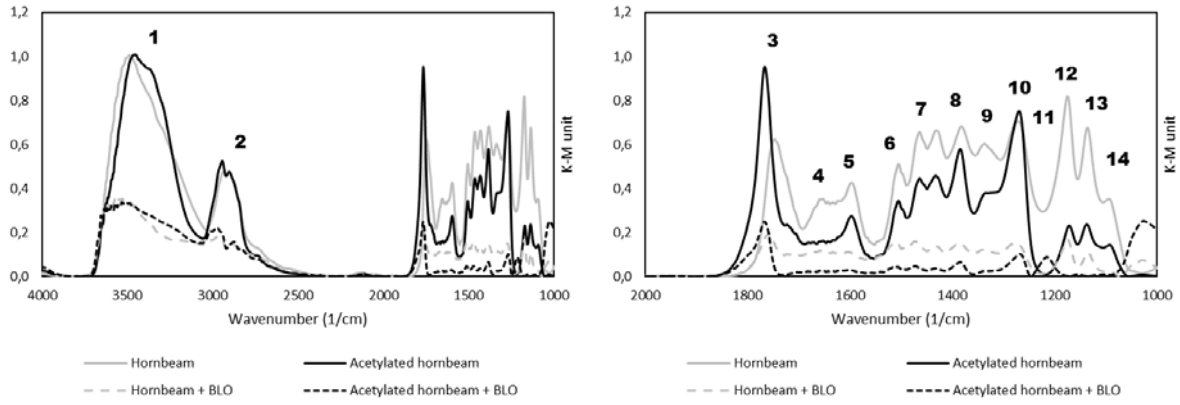

Fig. 6.
FTIR spectrum of hornbeam samples before 200 hour-long mercury-vapour lamp irradiation (BLO: boiled linseed oil-coated).

Lignin is responsible for retaining the wood strength and stiffness in case wood is contacted to moisture as lignin is the most hydrophobic component of the wood and it can limit the access of water (Winandy and Rowell 1984). During acetylation, in the acidic medium some parts of lignin can dissolve (Rowell 2005). The structural change and degradation of lignin was indicated by low absorption of aromatic functional groups (Fodor *et al.* 2017). As a result of mercury lamp irradiation the aromatic rings of lignin ruptured, which is indicated by lower absorptions in syringyl (5) and guaiacyl (6) lignin, asymmetric C-H deformation in lignin at 1476-1470 cm^{-1} (7), ring vibration in guaiacyl lignin (10) and aromatic C-H skeletal vibration (13). The absorption reduction in boiled linseed oil-coated samples is less notable.

Alterations of the lignin structure can also account for the slightly darker, walnut-like colour of acetylated hornbeam. These changes can include the oxidation of phenolic skeletal system as an effect of heat and acidic medium, as well as the reaction of lignin with evolving furfural in strong acidic medium (Dongre *et al.* 2015, Fodor *et al.* 2017) which results in not only structural changes of lignin, but also alterations in the colour of wood. However, the strength and stiffness properties of acetylated hornbeam increased (Fodor 2015) which indicate the degradation of lignin was not significant. The brightening of acetylated hornbeam during mercury lamp irradiation is probably associated with the extractive content. After acetylation, the extractive content of hornbeam increased (Fodor *et al.* 2017) which transformed during UV irradiation thus influence the colour. This effect was somewhat eased by coating with boiled linseed oil.

CONCLUSIONS

The aim of this work was to examine the effect of acetylation and boiled linseed oil coating on the photodegradation of hornbeam wood. 200 hour-long mercury-vapour lamp irradiation was carried out on hornbeam, acetylated hornbeam, boiled linseed oil-coated hornbeam and boiled linseed oil-coated, acetylated hornbeam samples.

According to our results, during UV irradiation hornbeam yellowed, the red hue (a*) and yellow hue (b*) increased. The dark, greyish brown colour of acetylated hornbeam brightened during UV irradiation because of the transformation of extractives. Unlike hornbeam, its brightness increased while the red and yellow hue decreased. Coating the samples with boiled linseed oil decreased the rate of colour change.

According to the FTIR spectra, lignin did degrade during mercury-vapour lamp irradiation. The absorption of functional groups in lignin decreased which led to the increase of methane, methylene, methyl and carbonyl groups. The rate of degradation and structural changes were highest in case of acetylated samples, but the strengthening polymers did not degrade notably.

In the future, other sealants and varnishes are to be tested on acetylated hornbeam that are natural, non-toxic and pigmented.

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EFFECT OF NATURAL AGEING IN INDOORS CONDITIONS ON THE COLOUR OF WOOD SURFACES FINISHED WITH NATURAL TRADITIONAL MATERIALS

Anca Maria VARODI
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: anca.varodi@unitbv.ro

Maria Cristina TIMAR
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: cristinatimar@unitbv.ro

Liu Xin YOU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: liu.xinyou@unitbv.ro

Camelia COJOCARIU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania

Abstract

This research paper is related to conservation - restoration of decorated furniture objects with intarsia by highlighting the influence of aesthetic quality of these special surfaces. The effects of natural aging by indoor exposure are presented.

Different wood species as well as finishing materials was employed in order to show in time evolution of chromaticity changes of the unfinished and transparently finished wood surfaces.

The effect of in time colour modification, after, a total period of 18 month of natural aging exposure was studied. Three wood species were selected ash (Fraxinus excelsior), walnut (Juglans regia) and Sycamore maple (Acer pseudoplatanus) and three type of traditional finishing material (beeswax, linseed oil, shellac). This traditional finishing material was the most used material in furniture manufactured of old objects.

The experimental results show that a protective finishing material resisted to light has to be used for maintaining the esthetical value of intarsia design. Otherwise the contrast colour between wood species is lost in time and will become null. The data are also useful for current approaches of the intarsia technique to provide data for an adequate choice of finishing technology with traditional products, which will add value to the objects.

Key words: *wood aging; colour stability; colour contrast; intarsia.*

INTRODUCTION

Colour, along with texture is an aesthetic attributes and elements that differentiate the wood species. Natural aging of materials is a progressive process (natural or artificial) that changes the original colour with an old look (http://manual.museum.wa.gov.au/book). This colour changes usually appearing as a darkening effect with different shade. The colour modification is depending of the wood species, some are become more yellowness, other more red and they are very distinctive compared to their original colour. The finishing material and environmental conditions are some important factors that influence the natural or artificial aging process (Timar et al. 2016, Liu et al. 2017, Reinprecht et al. 2017).

The natural colour of wood (veneer or timber), can give us information about wood species classification and can provide us some esthetical effects by intarsia design (Cismaru and Cismaru 2007, Unger and Wang 2009, Unger 2010). This type of decorating technics, dated for thousands years ago, is based on the colour contrast between wood species. Intarsia is an ornamentation technique that associates different wood species of different colours to achieve a high artistic effect (Wbee 1899, Caprara 1978, Tormey and Tormey 1982).The situation is even more complex in the case of historic furniture that is enriched with intarsia design. In this type of wooden piece several species are used in a single symbol, simultaneously exposed to the same conditions of

photodegradation. Wood aging is a very complex phenomenon due to a long and slow oxidation process, due to complex physical and chemical reactions and depends on the conditions in which the wood has been preserved, conditions that cannot be accurately known each time in the case of restoration (Unger and Unger 2010).

The different behaviour of the wood species needs to be very well known and understood. As a result of aging, the wood colour suffers changes over time. The same thing happens either outside or inside exposure. Although the inside exposure is not that aggressive, still sunlight (especially UV radiation) exposure through a window changes the wood colour.

Wood exposed under indoor conditions does not undergo colour changes due to UV radiation, because only a small part of it can penetrate the glass. In this case, the colour changes that occur are caused by visible light (400-700nm) that has enough energy to degrade the wood substances that give colour (http://www.fsec.ucf.edu/en/consumer/buildings/basics/windows/fading.htm, Liu 2017).

Visible light penetrates into the wood at 200μm deep, while UV radiation at 75μm, so light-induced modification is strictly a surface phenomenon. The rate of this colour change is usually dependent on: light intensity, wavelength, exposure time, wood species and environmental factors, of which humidity and temperature should be taken into account (Rowell 2015).

The present research is studying the colour modification and colour stability; of wood surface finished with traditional materials subjected to interior natural aging for a total period of 18 month. To this purpose, three wood species and three transparent finishing materials was taking in to account. The influence of this finishing materials is also a factor that contributing to the in time light resistance. Transparent finishing sometimes highlights the wood drawing and texture, as well as natural colour, following the decorative values of wood or veneers. Natural aging affects both the wood colour and the finishing film, so the initial colour contrasts changes over time.

OBJECTIVE

The main objective of the present research is the evaluation of chromaticity effects due to the indoor natural aging of wood unfinished and finished surface, based on the difference between wood colour and the concept of the comparative approach trough colour contrast.

MATERIAL, METHOD, EQUIPMENT

Wood material

Three wood species was selected (Fig. 1): ash –coded F (*Fraxinus excelsior*), walnut –coded N (*Juglans regia*) and Sycamore maple –coded P (*Acer pseudoplatanus*).

The wooden materials used in the present research have the dimension of 120mm (length) by 80mm (width) and 8mm (thickness), with radial faces. A number of 6 samples was selected, without any defects and coded. All the selected test samples were conditioned at 20ºC and 55% RH, in a climatic chamber, prior to finishing step.

Finishing material

As finishing materials was used beeswax (BW), linseed oil (LO) and shellac –coded SL being the most used finishing material in furniture manufactured of old objects. All this products are natural, traditional finishing materials. The sample was coded after finishing as well. As example, for ash sample finished with beeswax the final cod was F-BW, finished with linseed oil was F-LO and for the ash samples finished with shellac the final code was F-SL.

In the same context the unfinished ash surface was coded F-M, the walnut unfinished sample was coded N-M and unfinished sycamore maple sample was coded P-M.

For the beeswax preparation before application an amount of 50g of grinded wax was putted into a vessel and was heated a 100ºC (on top of a water bath); 250ml white spirit was added progressively until complete dissolution. Linseed oil was diluted for finishing, a dilution of 2.5:1 (linseed oil: white spirit) was made for the first and second layers applied on wood, whilst the final layers was applied non-diluted. For shellac preparation was used 100g shellac flakes and 10 g colophony (powder) dissolved under stirring in 1000ml ethyl alcohol on water bath.

Finishing wood surfaces

For a better understanding of how the natural aging affect the wood colour the study was performed on finished surface and on unfinished wood surface as well. After last sanded of wood with 240 grit size sandpaper and conditioned at the temperature of 20ºC and relative humidity of 55% the wood sample was manually finished.

The beeswax was applied with a soft pad covered with cotton in five layers, each of about 25g/m^2, applied by circular motions on the wood samples. The last step of finishing was the polishing of surface with a polishing machine equipped with a felt.

Linseed oil was applied with a brush in five layers, each one of about 125g/m^2. Between layers the finishing surfaces was sanding with 360 grit size sandpaper**.** For the first two layers a diluted linseed oil solution in white spirit (as it mentioned above) was used and for the last three layers undiluted linseed oil was applied.

Shellac finishing was made first by brush in three successive layers of about 110g/m^2 at intervals of 2-4 hours (drying time) without intermediary sanding. After the formation of base coat, the finished wood samples were sanded with 360 grit size sandpaper. In order to obtain a high gloss surface, it was considered necessary to filling the pore employing a polishing with cotton wool until a satisfactory degree of structure filling were obtained.

The drying interval between layers was 24 hours at environmental temperature.

Natural ageing test

The natural ageing test in indoors conditions at room temperature under the influence of natural light filtered by window glass was carried out in the L5 laboratory at the CDI Institute of Transilvania University of Brasov. Samples were exposed vertically to sunlight in a test rack located near the window, facing the South direction at a height of 650 to 1350mm from the ground level (Fig. 2). The testing period was about 18 months started at 1 May 2015 and ended at 1 November 2017. The wood samples exposed to natural ageing were covered with black cardboard on half of their surface; this aspect is visible in Fig. 2.

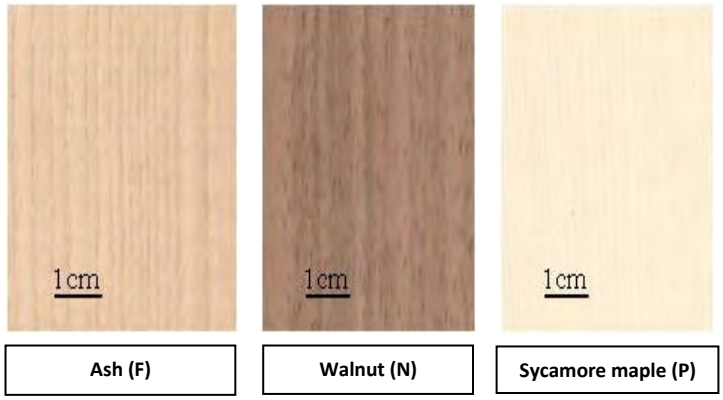


Fig. 1.
Wood sample used in present research (initial scanned images).

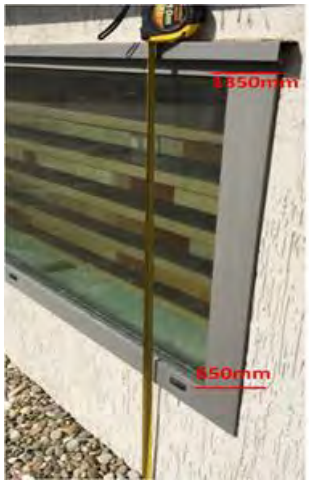


Fig. 2.
Wood sample exposed at CDI - Institute (exterior view).

Principle of method

The diagram presented in Fig. 3 shows a simulated in time colour changes and colour contrasts of natural ageing exposure, on two hypothetic type of sample with initial contrast white and grey.

In the top of the scheme is presented de initial state, according to the adjacent scale of colour. In this case, we have an initial contrast CI = 1.

In the first case, after time passing, the booth initial colour of sample suffering changes and the contrast value remaining the same C = 1. If we consider these contrasts in wood furniture intarsia design, we will have a similar contrast, so the colour changes will not being affect, the obvious colour effect remains.

In the case 2, the initial colours are changed different and the contrast is not the same we will have a value C = 2. In this case the contrast will increase. This can be a positive effect, but also a negative one because of the high difference between species, the esthetical value brought by the intarsia design will be lost.

In case 3, we will have also different colours changes. The value of contrast is in this case C = 0, so the desired colour effect will be null. The intarsia design, which is generated by the differences between colours, is not visible.

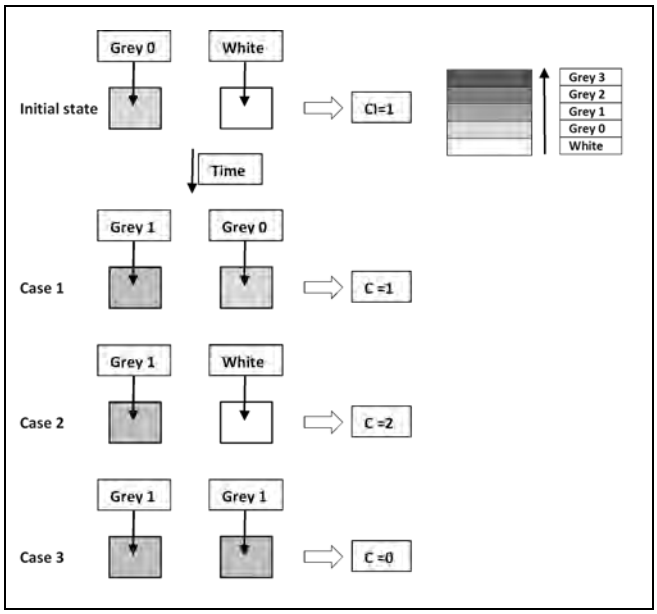


Fig. 3.
The scheme of simulated colours changes of two hypothetic type of sample.

Based on this principles in the present paper will be determined the effect of natural ageing on simulated intarsia using the three studied wood species. In Fig. 4 is presented the scheme of this simulated intarsia.

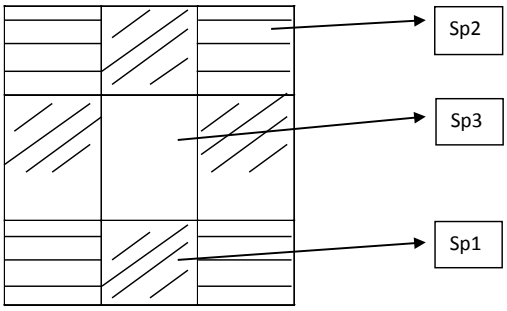


Fig. 4.
The scheme of simulated intarsia made of three wood species (Sp. 1, 2, 3).

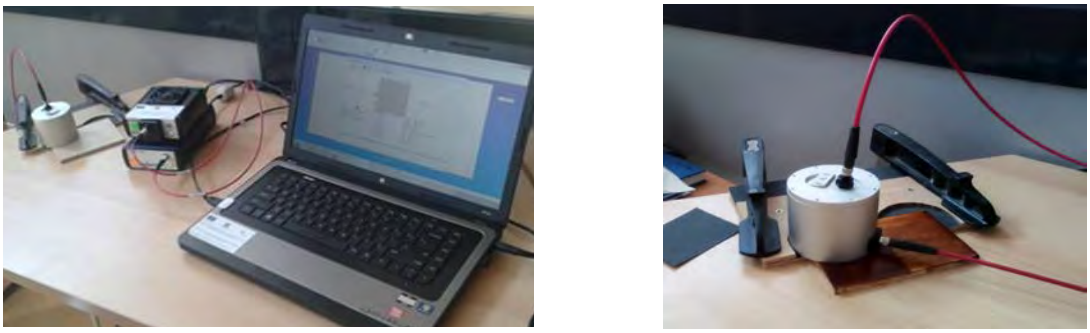
For this propose the wood samples and finished surfaces were scanned by a HP LaserJet Pro CM1415 colour multifunction printer.

Colour measurements

Colour measurements were performed in the CIE-Lab system (Fig. 5a) employing a spectrometer AvaSpec-USB2 equipped with an integrating AVA sphere having a diameter of 80mm, interconnected by optical fibbers, provided with dedicated software.

All colour measurements were performed for each test sample in 4 points, before testing – month 0 (L0) and at different interval of time (month 1, 2, 3, 4, 5, 6, 8, 10, 12, 18). The measurement was performed on finished surface and on unfinished one. For each type of sample it was calculated an average of 24 points.

In order to repeat the measurements in the same areas, a sample fixing plate (Fig. 5b) was designed and manufactured. This allows reproductive colour measurements in terms of investigating the same areas for unfinished and finished wooden samples, before and after ageing. Otherwise, the uniformity of wood colours and wood colours changes will have a higher dispersion of the experimental values.



a b

Fig. 5.
AvaSpec-USB2 spectrometer: a) General view on components; b) Integrated AVA sphere and the fixing plate for reproductive colour measurement.

The colour changes was calculated for each colour coordinate (*L**, *a** and *b**) as related to its initial value on the same sample and in the same point. Finally, the total colour change (Δ*E**) was calculated in each point, according to equation (1):

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

where:

Δ*L** is the luminosity change after aging compared with initial value:

$$\Delta L^* = L^*_{\text{exposed}} - L^*_{initial} \quad (2)$$

Δ*a** is the redness change after aging compared with initial value:

$$\Delta a^* = a^*_{\text{exposed}} - a^*_{initial} \quad (3)$$

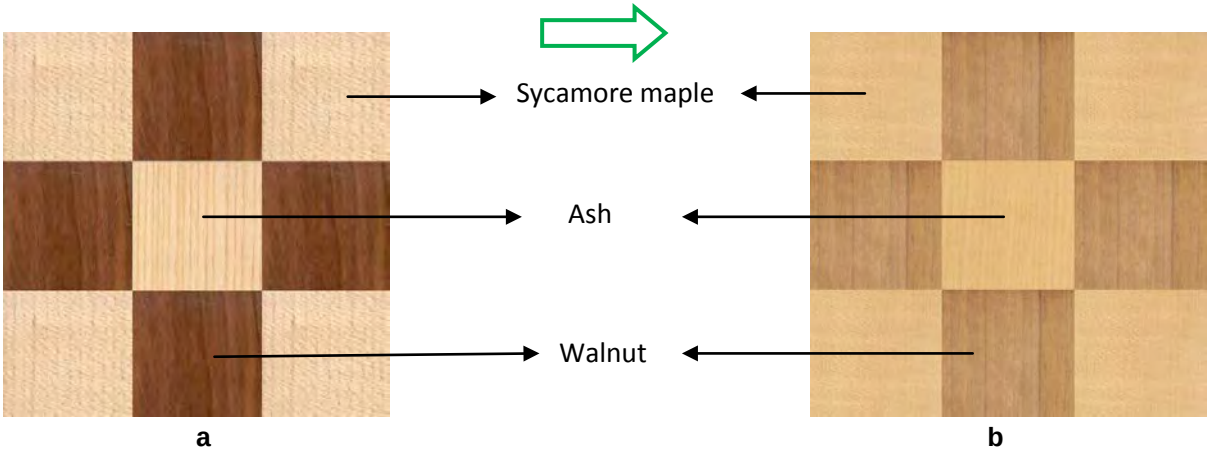
Δ*b** is the yellowness change after aging compared with initial value: :

$$\Delta b^* = b^*_{\text{exposed}} - b^*_{initial} \quad (4)$$

RESULTS AND DISCUSSION

Visual aspects

In Fig. 6 are presented the scanned images of simulated intarsia using the three wood species studied finished with beeswax, as example, recorded in initial state compared with the natural aged images scanned after 18 month exposure.



a b

Fig. 6.
Comparative view of the scanned images of simulated intarsia between initial state (a) and after 18 month of natural aging exposure for all three studied wood species.

It can be observed that the colour intensity is diminished and the contrast between species is almost lost. If we compare ash wood with sycamore maple it can be said that the colour contrast is

lost. This behaviour is the same like case 3 of principle contrast changes, which is explained above when the value of contrast is considered null.

The wood surfaces underwent gradual colour changes; the contrast was changed within the time. Referring to the natural ageing these slow changes is expected, in the first period of time (in the first six month) the colour changes is more rapid but in time the effect of aging is slower. For a better view of the colour and contrast modification for all samples, finished and unfinished, in Fig. 7 are presented the simulated intarsia images. These images include only first six month (1, 2, 3, 4, 5, 6) and the last month (18).

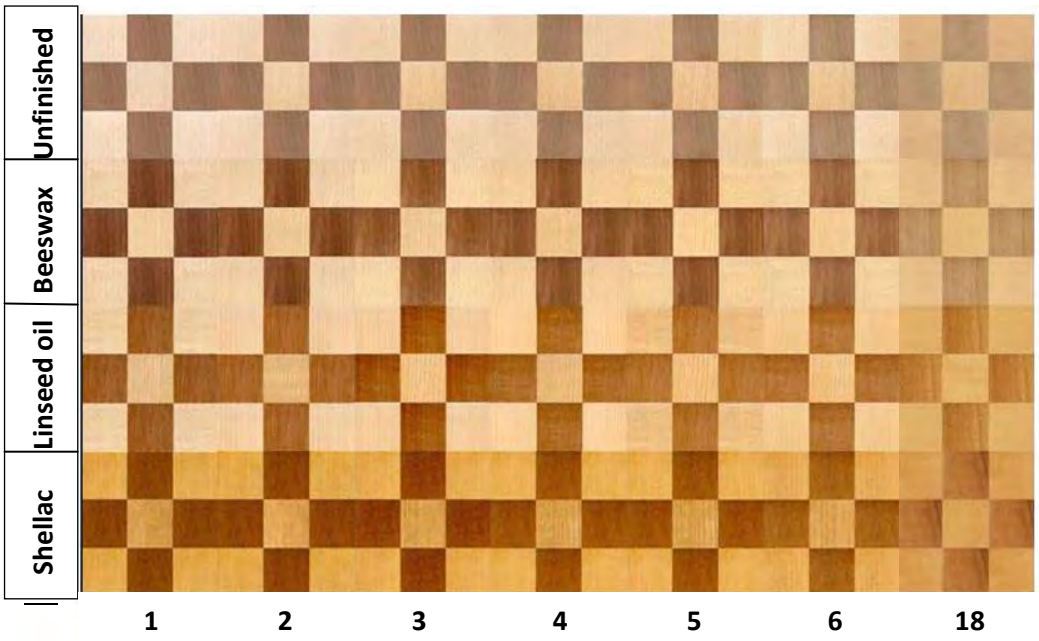


Fig. 7.
The simulated intarsia images for month 1, 2, 3, 4, 5, 6 and the last month 18.

Analysing the images shown in this figure (Fig. 7) it can be said that the colour differences between the studied wood species begin to fade, for all finished or unfinished surface, indifferent of the finishing material. However, the most affected wood surfaces are unfinished one, which leads to the idea that the finishing materials protect the surfaces.

Regarding to the finished surfaces only, the most durable finish material who preserve best the contrast is linseed oil.

Colour Changes

The visual aspects presented above can be analysed quantitatively by colour measurements in the CIE-Lab system. In the figure 8 are the graphical representations of in time evolution of colour coordinates (lightness, redness and yellowness) for all three woods samples unfinished (P-M, N-M and F-M) and finished with all finished material studied.

By analysing this images, presented in figure 8, it can be stated that all wood species suffered a progressive colour change, mostly in the first six month as was previously observed.

The lightness is decreased for sycamore maple and ash for unfinished and finished surface for all finishing material studied. Walnut registered an increase of lightness in all period of time exposure for all woods sample unfinished and finished.

In generally is observed an increase of redness. Regarding to yellowness it can be observed that at first an increase of this light coordinated is registered and after 12 month this trends stops and is almost linear or registered an decreasing.

Though for all wood species the increasing or decreasing of colour coordinates was not linear and not similar between wood species. The same behaivour was observed indifferent of the finishing material.

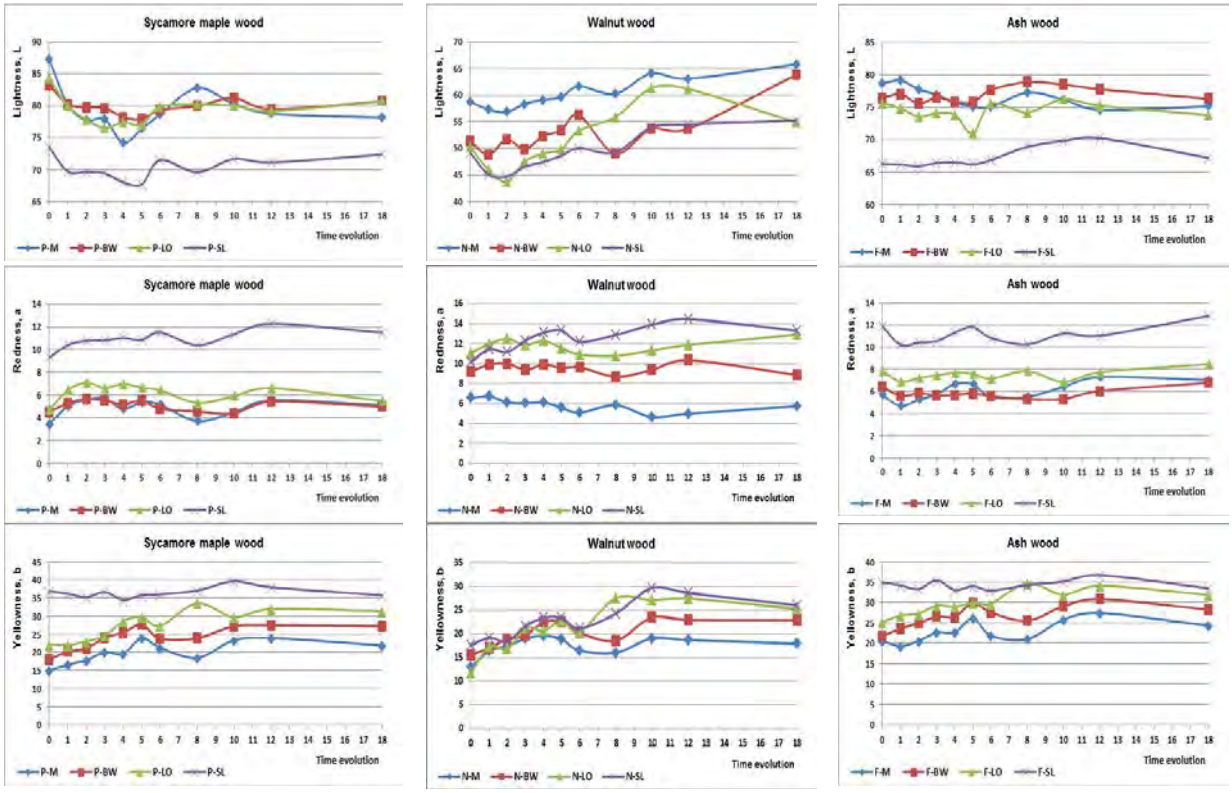


Fig. 8.
In time evolution of colours coordinates: lightness (L –top), redness (a–in the middle) and yellowness (b-bottom) for all wood species unfinished and finished.

Contrast Changes

As was stipulated before al visual colour changing can be analysed quantitatively by colour measurements.

In the same context, it can be calculated the contrast modification in time between species. The in time contrast modification is presented as calculated colour differences between wood species.

In Table 1 are presented the data for contrast modification between ash and sycamore maple wood (ash-considered the darker species versus sycamore maple-considered the lighter species).

Table 1

Colour contrast (differences) between ash and sycamore maple wood

Time	Colour contrasts between Ash and Sycamore maple											
	M-unfinished			BW			LO			SL		
	ΔL	Δa	Δb	ΔL	Δa	Δb	ΔL	Δa	Δb	ΔL	Δa	Δb
0	-8,58	2,24	5,67	-6,81	1,94	3,86	-8,65	3,19	3,03	-7,23	2,59	-1,95
1	-1,04	-0,31	2,61	-3,23	0,30	3,33	-5,13	0,47	4,81	-3,52	-0,15	-2,00
2	0,03	-0,32	2,78	-4,18	0,19	3,89	-4,28	0,11	4,26	-3,74	-0,35	-1,90
3	-0,95	0,05	2,60	-3,09	0,12	2,46	-2,44	0,85	4,63	-2,99	-0,25	-1,16
4	1,53	1,86	3,04	-2,32	0,50	1,07	-3,56	0,71	0,36	-1,47	0,32	-1,41
5	-2,26	1,27	2,23	-2,06	0,30	2,21	-5,05	0,95	0,14	-1,49	0,98	-1,76
6	-3,54	0,37	0,59	-1,61	0,79	3,68	-4,27	0,75	2,45	-4,62	-0,66	-3,07
8	-5,53	1,80	2,59	-1,06	0,77	1,63	-5,99	2,52	1,20	-0,70	-0,13	-2,80
10	-4,07	1,92	2,47	-2,69	0,95	1,85	-3,74	0,94	2,26	-1,86	-0,10	-4,45
12	-4,15	1,77	3,51	-1,68	0,62	3,39	-3,88	1,12	2,18	-0,86	-1,22	-1,28
18	-2,96	1,94	2,43	-4,36	1,78	1,09	-6,88	2,98	0,53	-4,17	1,33	-2,24

Analysing the data presented in Table 1 it can be seeing that the contrast between wood species is decreasing and a possible esthetical effect is being faded. The lightness contrast is reduced

more for unfinished sample, that for finishing one. The protection generated by the finishing material is visible here as well. The most affected contrast referring to lightness of finished sample was registered for beeswax (from -6.81 to -4.36) and shellac (from -7.23 to -4.17). The most resisted sample is the wood surface finished with linseed oil (from -8.65 to -6.88). In generally is observed a slightly modification of redness and the yellowness difference is decreasing.

As previous was showed that the increasing or decreasing of colour coordinates was not linear the effect is similar for the contrast difference between wood species. This behaviour is better observed in figure 9 in which is presented the global colour modification between ash and sycamore maple.

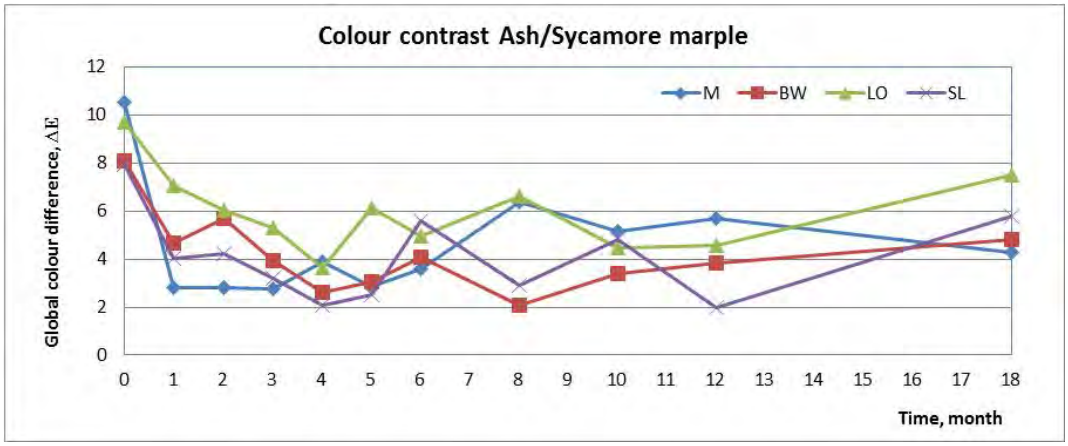


Fig. 9.
Global colour difference as colour contrast between ash and sycamore maple wood.

The graphical representation showed that for all unfinished and finished surfaces the contrast decreased with the exposure time. The recorded data show that there is a clear evolution of the species contrast between the first six months, after the wood species aged differently so we cannot say that there is a clear evolution. However, there is no longer the same contrast between species, since it is clear that wood species change their colour during aging, even for indoor exposed.

The in time evolution of contrast modification between walnut and sycamore maple wood (walnut-considered the darker species versus sycamore maple-considered the lighter species) is presented in table 2.

Table 2
Colour contrast (differences) between walnut and sycamore maple wood

Time	Colour contrasts between Walnut and Sycamore maple											
	M-unfinished			BW			LO			SL		
	ΔL	Δa	Δb	ΔL	Δa	Δb	ΔL	Δa	Δb	ΔL	Δa	Δb
0	-28,49	3,10	-1,88	-31,86	4,67	-2,37	-34,01	6,31	-10,45	-24,33	0,88	-19,27
1	-22,91	1,70	-0,32	-31,49	4,61	-3,25	-34,04	5,54	-4,67	-24,61	1,01	-17,07
2	-20,92	0,54	-0,36	-28,08	4,36	-2,15	-34,12	5,36	-6,23	-24,98	0,37	-16,77
3	-19,55	0,35	-1,08	-29,76	3,85	-4,43	-29,02	5,21	-3,31	-22,88	1,50	-15,02
4	-15,09	1,33	-0,03	-25,91	4,71	-2,95	-28,37	5,30	-7,98	-20,62	2,09	-11,09
5	-16,79	0,20	-5,02	-24,53	4,03	-5,08	-27,23	4,86	-7,02	-19,11	2,49	-12,39
6	-17,01	-0,07	-4,81	-23,07	4,83	-3,63	-26,48	4,45	-6,92	-21,47	0,69	-14,87
8	-22,53	2,12	-2,46	-30,92	4,11	-5,33	-24,33	5,42	-5,98	-20,38	2,49	-12,76
10	-16,14	0,11	-4,33	-27,48	4,98	-3,72	-18,68	5,36	-2,55	-17,68	2,55	-9,90
12	-15,67	-0,59	-5,25	-25,83	4,92	-4,53	-17,97	5,23	-4,47	-16,64	2,17	-9,35
18	-12,37	0,64	-4,00	-16,84	3,85	-4,29	-25,84	7,40	-6,05	-17,12	1,83	-9,74

Analysing the data presented in table 2 it can be seeing that the contrast is modified in this case to. The lightness contrast is more affected for unfinished sample, that for finishing one, as was in previous situation and is decreasing from -28.49 to -12.37. The protection generated by the finishing material is visible here as well. The most affected contrast referring to lightness of finished sample

was registered for beeswax and linseed oil. The most resisted sample in this case is the wood surface finished with shellac, the lightness decrease from 24.33 to -17.12. The redness increase for unfinished surface and for wood surface finished with beeswax, and increase for linseed oil and shellac. The same behaviour is registered for yellowness.

In the Fig. 10 we can observe as graphical representation the evolution of the chromatic contrast between walnut considered the darker species and sycamore maple considered the lighter species.

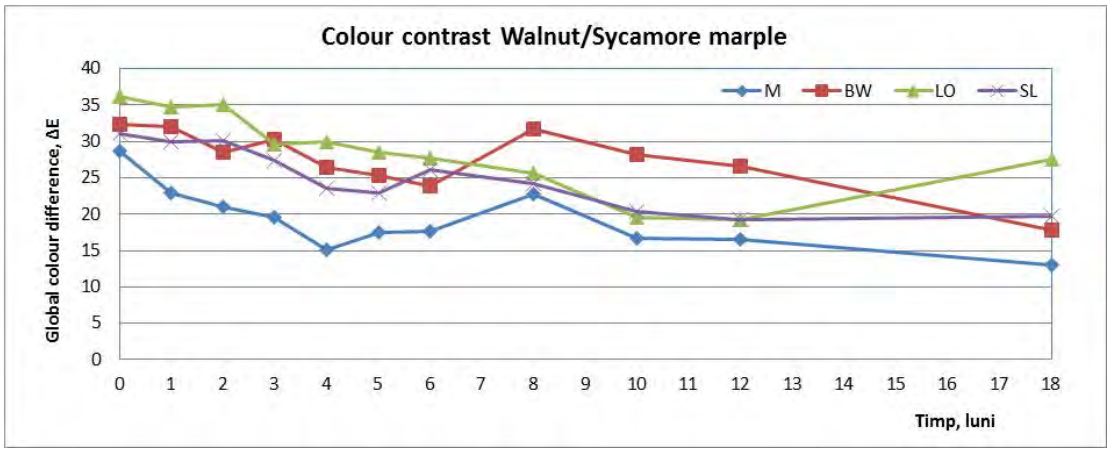


Fig. 10.
Global colour difference as colour contrast between walnut and sycamore maple wood.

By analysing the graphic representation it can be stated that in all cases the evolution of the chromatic contrast is the same. It decreases significantly in the first exposure period, and then varies, because of the wood surfaces resistance to light, having different properties.

In this case the data show that there is a clear evolution of the species contrast between the first six months as well, but after this period the evolution continues for wood unfinished sample and for finished one with beeswax and shellac.

CONCLUSIONS

Environmental factors, UV radiation from the natural light spectrum, cause aging of materials affecting both woods, finishing materials and finishing surfaces. The colour changes is the most visible and sensitive indicator of aging phenomena.

The aging of the finished surfaces is a cumulative result of wood substrate behaviour and of finishing films behaviour, the concrete effect being influenced both by the wood species and by the type of finishing material.

The results obtained within the present research demonstrated that ageing induced specific macroscopic aspect related most in colour changes. Natural light caused colours changes at a slower rate, depending of the wood species and on the finishing material.

All unfinished surface are more sensitive that finished one. The finishing materials generate a protect effect of wood surfaces against aging. The finishing material that is most resisted to natural ageing exposure is linseed oil, indifferent of the wood species.

As result, aging affects the aesthetics surfaces decorated by intarsia as a result of the modification of the original colour contrasts. This effect is the unmistakable patina of old wood objects, which generally have aesthetic value.

However, after a certain degree of aging, the colour suffers changes and it can be too pronounced, or can fade to a level that no longer highlights the decoration made by intarsia. In this case, we are talking about the aging patina that needs to be diminished by restoration action.

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PREPARATION OF BIO-OIL-PHENOL-FORMALDEHYDE RESIN: PROPERTIES AND BONDING PERFORMANCE OF RESIN

Caglar CEKIC
Karabuk University, Faculty of Forestry
78050 Karabuk, Turkey
Tel: +90 (370) 433 86 31, E-mail: caglarcekic@karabuk.edu.tr

Günay ÖZBAY
Karabuk University, Faculty of Engineering
78050 Karabuk, Turkey
Tel: +90 (370) 433 86 31, E-mail: gozbay@karabuk.edu.tr

Erkan Sami KOKTEN
Karabuk University, Faculty of Engineering
78050 Karabuk, Turkey
Tel: +90 (370) 433 20 21, E-mail: erkansamikokten@karabuk.edu.tr

Ayhan OZCIFCI
Aksaray University, Faculty of Engineering
68100 Aksaray, Turkey
Tel: +90 (382) 288 23 00, E-mail: ayhanozcifci@aksaray.edu.tr

Abstract

The purpose of this study was to demonstrate the performance of bio-based resins (BPF) designed as adhesive. In this way, the bio-oil was incorporated into phenol formaldehyde resin (PF) at 50%wt. phenol replacement level. The physical properties of the resins were determined. The bonding performance of wood specimens bonded with BPF resin were tested under dry condition. The bonding performance of the samples was comparable to those of wood specimens bonded with commercial PF. The results demonstrated that the bonding performance of the BPF resin was slightly lower than that of commercial PF resin. Bio-oil can be directly used as a chemical feedstock for the synthesis of wood adhesive production.

Key words: *adhesive; bio-oil; phenol formaldehyde; shear strength; wood.*

INTRODUCTION

Last decades researchers have been focused on replacement of part of the fossil fuel consumption and chemical production by renewable energy sources. Biomass, the most important energy source in several developing countries, can be converted to useful products by thermo-chemical processes or bio-chemical processes. There are several thermo-chemical conversion routes of biomass, such as pyrolysis, gasification, and combustion (Centi et al. 2013, Goldemberg and Coelho 2004, Goyal et al. 2008, Balat et al. 2009).

Pyrolysis is thermal decomposition occurring in the absence of oxygen. Three end-products occurred in solid, liquid (bio-oil) or gas phases with different conditions depending on the operation conditions. Pyrolysis for liquids production is currently of particular interest as the liquid can be stored and transported, and used for energy, chemicals or as an energy carrier. Chemically, bio-oil is mainly composed of levoglucosan, furfural, phenol and aldehyde. Phenolic compounds accounted for the largest amount in the bio-oil. They are valuable chemicals and can be used as intermediates in the synthesis of pharmaceuticals, for the production of adhesives and the synthesis of specialty polymers (Bridgwater 2012, Özbay et al. 2015, Žilnik and Jazbinšek 2012, Özbay 2015, Ziolek 2014).

Phenol formaldehyde (PF) resol adhesives have been widely used as wood adhesives for production of many wood composites, such as plywood, oriented strand board and laminated veneer lumber (LVL), due to their excellent bonding performance, water resistance and durability. They are commonly synthesized using petro-chemicals such as phenol and formaldehyde. However, increases in petroleum prices, along with formaldehyde emission concerns and general phenol safety issues. There are extensive researches on replace phenol with renewable resources (Pizzi 1983, Zhang et al. 2013, Wescott and Frihart 2004).

OBJECTIVE

In this work, the bio-oil was incorporated into phenol formaldehyde resin (PF) at 50% wt. phenol replacement level. The physical properties including pH, dynamic viscosity, gel time and solid contents of the resins were determined. The shear strength test was conducted on the connection surface of samples jointed by the bio-based PF resin. The bonding performance of wood specimens bonded with BPF resin were evaluated under dry conditions.

MATERIAL, METHOD, EQUIPMENT

Bio-oil

Bio-oil obtained from Karabuk University, Forest Faculty Research Laboratory, was recovered during vacuum pyrolysis of pine wood sawdust. Main properties of the bio-oil were given in Table 1. The bio-oil consisted in oxygenated organics, such as, aldehydes, ketones, phenols, benzenes, alcohols and polycyclic aromatic hydrocarbon (PAH). Phenols were the dominant component in the bio-oil (Özbay et al. 2016).

Table 1

Main properties of the bio-oil

Solids (wt%)	<1
Viscosity (cSt @ 40 _C)	54,6
Ash (wt%)	<0,3
C (wt%)	62,39
H (wt%)	7,14
O (wt%)	30,47

Chemicals

A commercial FP resin was tested in this work as reference resin. The commercial PF adhesive was supplied by POLİSAN chemical company in Izmit, Turkey. Chemicals (e.g., phenol and formaldehyde), supplied by GENTAŞ chemical industries, İzmit, Turkey. Sodium hydroxide (NaOH) was purchased from Sigma-Aldrich and used without further purification.

Synthesis of the resins

Firstly, an adhesive reactor was charged with 500 g of the bio-oil and 590 g formaldehyde. The reaction temperature was raised up to 60 °C within 30 min. and then 50 g of NaOH solution (50% wt.) (1/3 of total NaOH weight) was added to the reactor. The mixture was stirred and heated to 90 °C, afterwards kept at that temperature for 30 min. Then the reactor was cooled to 60 °C again. Secondly, 500 g phenol and 1000 g formaldehyde were added in the reactor. The reaction temperature was raised up to 70 °C within 15 min., followed by the addition of the remaining 2/3 of NaOH. Finally, the reaction mixture was stirred and heated to 90 °C, then kept at that temperature for 60 min. After the reaction, the system was cooled down to room temperature.

Resin characterization

The pH and viscosity of the resins were measured with a pH meter (TES-1380 pH meter) and a viscometer (Brookfield digital viscometer, model: Dv-IPrime) at 25 °C respectively. Gel time was determined by charging 2 g of the resin into a test tube and heating the test tube in a bath at 100 °C. Gel time was defined as the time period from the immersion of the test tube into the bath to the beginning of the resin gelation. The solid content measurement was according to ASTMD 3529.

Preparation of wood samples for shear strength test

The beech wood (*Fagus orientalis* Lipsky) wood was chosen randomly from a commercial timber company in Karabuk, Turkey. The special emphasizes was put on the selection of wood material. The selected specimens were cut at the sizes of 50×100×200 mm. The samples were conditioned in a climate room at 20 °C and 65% relative humidity and allowed to reach a nominal equilibrium moisture content of 12%. Then, they were re-sized to relate to the standard (BS EN 205). The dimensions and shape of the samples were given in Fig. 1. The bonding test samples were bonded with the reference and BPF resins, which were applied at rate of about 180 g/m^2 on single bonding surface according to advice of manufacturer. The measurements of compression and bonding

strength tests were carried out in a Zwick/Roel Z50 universal test machine with a testing speed of 1 mm/min.

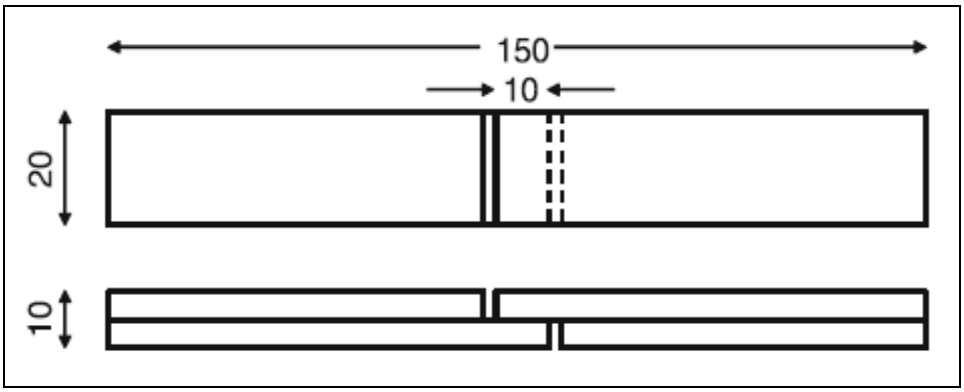

Fig. 1.
Shear strength test sample (size given in mm)

RESULTS AND DISCUSSION

Main properties of the resins

The properties of the resins were given in Table 2. The pH values for the bio-oil-PF resin and the commercial PF resin were similar. The viscosity of the bio-oil-PF resin was a bit lower than the viscosity of the commercial PF resin. This result can be explained by the fact that the viscosity of the bio-oil PF resin was continuously adjusted during the synthesis process. The bio-oil-PF resin had a much shorter gel time than the reference resin (as shown in Table 1). The solids content of bio-oil-PF resin and commercial PF resin were 45,71% and 48,78%, respectively. The solids content of the bio-oil-PF resins was lower than the solids content of the commercial PF resin. Zhao and co-workers noticed that commercial PF resin generally contains a high amount of urea (Zhao et al. 2010).

Table 2

Main properties of the PF resins

Properties	Reference resins (Com. PF)	Bio-oil-PF resins (50% wt. bio-oil)
pH (20 °C)	11,90	11,82
Viscosity (25 °C, cPs)	310	332
Gel time at 100 °C (s)	175	127
Solids content (%)	48,84	46,76

Shear strength of the resins

The bonding performance of the wood samples bonded with the bio-oil PF resin and commercial PF resin were shown in Table 3. Bio-oil-PF resins with 50% phenol replacement had similar bonding strengths to the commercial PF. The bonding performance of the bio-oil PF resin was slightly lower than that of commercial PF resin. Relating to the standard, samples of wood bonded with the bio-oil PF resins obtained the requirements for durability requirements (at least 10 N/mm^2). These findings were confirmed by Sukhbaatar et al. 2009.

Table 3

The bonding performance of the wood samples

	Resin type	
Shear strength (N/mm2)	**Reference resins (Com. PF)**	**Bio-oil PF resins (50% wt. bio-oil)**
Minimum value (N/mm2)	8.43	7.94
Maximum value (N/mm2)	12.80	12.57
Average value (N/mm2)	10.62	10.15
Std. Dev.	1.03	1.08

CONCLUSIONS

In this work, the bio-oil was used to substitute phenol up to 50% wt. in the synthesis of bio-based PF resin. The bonding performance of the bio-oil PF resin was slightly lower than that of commercial PF resin. The bonding strength of bio-oil PF resin was highly comparable with that of commercial PF resin. Bio-oil can be directly used as a chemical feedstock for the synthesis of wood adhesive production.

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EUROPEAN CO-OPERATION IN WOOD RESEARCH FROM NATIVE WOOD TO ENGINEERED MATERIALS: PART 1: CHEMICAL MODIFICATION WITH NATIVE IMPREGNATION AGENTS

Peter RADEMACHER[1], Róbert NÉMETH[2], Miklós BAK[2], Fanni FODOR[2], Tamás HOFMANN[3]
University of Sopron, [1]EFKK Res. Center for Forestry and Wood Industry/ [2]Simonyi Karoly Faculty of Engineering, Wood Sci. and Applied Arts, Inst. of Wood Sciences/ [3]Fac. of Forestry, Inst. of Chemistry
Bajcsy-Zs. u. 4, H-9400 Sopron, Hungary
Tel.: +36-99 518 769, Fax: +36-99 312 240, Email: Peter.Rademacher@uni-sopron.hu

Jan BAAR, Petr PAŘIL, Radim ROUSEK, Zuzana PASCHOVÁ, Pavel SABLIK
Mendel University, Faculty of Forestry and Wood Technology, Department of Wood Science
Zemědělská 1, 613 00 Brno, Czech Republic
E-mail: Jan.Baar@mendelu.cz

Gerald KOCH, Uwe SCHMITT, Dietrich MEIER, Yongshun FENG, Eckhard MELCHER, Bodo SAAKE[*]
Thünen Institute of Wood Research/ [*]University of Hamburg, Center for Wood Science
Leuschnerstr. 91d, 21031 Hamburg, Germany, Email: gerald.koch@thuenen.de

Milan ŠERNEK
University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology
Rozna dolina, c. VIII/34, 1000 Ljubiljana, Slovenia
Email: milan.sernek@bf.uni-lj.si

Abstract

The paper presents the results of a German – Czech – Hungarian –Slovenian wood research consortium, dealing with wood modification techniques by using renewable modification agents for the outdoor use of local, originally non-durable modified wood species like beech, poplar and pine sapwood. Higher value assortments of naturally durable and dimensional stable wood species, like oak and black locust from European forests, or others from tropical/sub-tropical forests are limited and partly criticized due to their non-sustainable production and harvesting conditions. Other reasons are the usage of non-local wood sources or the biocide treatment of non-durable species which have a higher impact on the environment because of long distance transport and the use of environmental critical chemicals. Due to simultaneously limited raw material volumes from fossil origin and their negative impact on climate and environment, the production and application of sustainable and renewable materials, like wood or further biomass assortments, become more and more important. Not only the sustainable production and use of the CO_2-fixing material WOOD itself, but also the production and utilization of renewable liquid agents for the impregnation stage in the wood modification process-chain as well as the finishing or gluing process-steps are additionally helpful to improve the sustainability of the industrial production.

The results show, that extracts from plant or tree residues with natural biocide or cross-linking behaviour as well as heat-treatment process residues of biomass materials, like collected liquids from thermal modification processes or hydro-thermal carbonised as well as pyrolysed or liquefied biomass or wood residues can result in a distinct improvement on the wood properties. These enhanced wood qualities enable the outdoor use of originally non-durable or non-dimensionally stable wood assortments.

Key words: *wood modification; renewable impregnation agents; outdoor wood applications; dimensional stability; durability.*

INTRODUCTION

The production and application of the sustainable and renewable material WOOD under local conditions and its higher value use under outdoor conditions have to be evaluated under following background conditions:

- Climate change, global warming, CO_2 re-emission from fossil sources

- Forest conversion, limited high value wood assortments, non-sustainable produced tropical wood assortments or biocide treatment as well as plastic, metal, or concrete materials
- Renewable, sustainable and locally produced wood, environmentally friendly chemical modification treatments (CMT) & treatment agents (impregnation, finishing, adhesive)
- Research interactions in EU-Networks (COST FP1407, DE-CZ-HU-SE-SI-NET, DANUBE-Network)

OBJECTIVE

The objective of the present research was to evaluate the possibilities of both, the utilization of non-durable local wood species as well as their property improvement for outdoor applications by wood-modification agents from native, renewable origin.

SHORT STATE-OF-THE-ART OF CMT AND EXPECTED MATERIAL PROPERTY CHANGES

During the last decades the production and utilization of renewable biomass from forest, agriculture and fast growing trees in short rotation plantations (SRPs) increased and are important for the future biomass supply with also options for different usages. Reasons for this are: 1) enhanced usage or even over use of traditionally managed forests (Mantau and Bilitewski 2005); 2) decreasing storage of fossil raw materials and energy sources (FAO 2005); 3) increasing amount of carbon dioxide in the atmosphere and global warming due to burning of fossil carbon sources (Cox et al. 2000); 4) need of higher application of more and climatically better adapted tree species under changing climate conditions (Blohm et al. 2014).

During the development of wood cell walls until maturation and functionality of the cells, wall architecture and chemical composition become distinctly changed (Zimmermann and Brown 1980; Fengel and Wegener 1989; Wagenführ 1999; Faix 2008). Secondary changes, especially in case of durable heartwood, occur at the transition from sapwood to heartwood by the incorporation of phenolic compounds in the carbohydrate-lignin complex (Hillis 1962; Klumpers et al. 1994; Faix 2004). During subsequent aging of cell walls, additional organic components (Gierlinger et al. 2003; Haupt et al. 2003; Grabner et al. 2005) or mineral elements can be bonded on or included in fibril-structure of wood cell walls (Rademacher et al. 1986; Faix, 2004), leading to both increased durability and dimensional stability of wood. Also wounding (Rademacher et al. 1984; Shigo 1986; Frankenstein and Schmitt 2006), emissions (Hapla 1992) or climate stress (Schweingruber 1993) can initiate tertiary structural or chemical changes in cell walls. The knowledge about these basic mechanisms has been strengthened due to many investigations in the past (see overviews of Schwager and Lange 1998, Faix 2008, Wagenführ and Wagenführ 2008, Schmitt and Koch 2009), mainly dealing with most utilized tree species like spruce and pine or beech and oak. However, the knowledge about structure and chemistry of lesser used wood species or even the mechanism and process of technical or biotechnical changes in cell walls due to wood modification – leading to an improvement of properties – is still not fully understood (Zimmermann and Brown 1980; Ermeydan et al. 2012).

The improvement of wood properties by wood modification (Hill 2006; Militz and Mai 2008) became a more and more important goal and suitable methods have been developed for innovative and additional use of wood as a renewable raw material (Klemm et al. 2005). The transformation of our knowledge about mechanisms and processes of modification from technical (Militz et al. 1997) to small-scaled cellular (Mahnert et al. 2013) or molecular level (Grabner et al. 2005) could help to deepen our understanding and enlarge our instruments to establish new materials (Meier et al. 2001; Heiduschke and Haller 2010).

A lot of efforts have been done in the past to improve the properties of wood by chemical or mechanical treatments (Rowell 1983; Wepner and Militz 2005; Hill 2006), but the cellular or even sub-cellular mechanisms (mode of action) behind this are more or less unclear (Zimmermann and Brown 1980; Ermeydan et al. 2012). The following results deal with the exposure of basic mechanisms which lead to higher density, higher strength, higher dimensional stability and higher durability of wooden material. Collecting information on the microscopic structures as well as the chemical composition of cell walls before and after modification treatments is essential to understand the mechanisms behind changed 1) cell wall stability, 2) cell wall penetrability and 3) cell wall impregnation.

METHODS

A set of renewable agents for wood impregnation was developed for the following material-resources and processes (see also Table 1):

- Production of liquid residues from thermal treatment (TT), Hydro-Thermal Carbonisation (HTC), pyrolysis-processes (Pyrol), Liquid Wood (LW) and Robinia extracts (RobExtr); see Table 1.

- Impregnation of beech, poplar and pine sapwood samples with these liquids (using vacuum 20 kPa/1 hour) under calculation of the weight-percent-gain (WPG) and leaching tests, using 10 cycles to yield the weight-percent-remain (WPR) of the non-leached fraction.
- Concentration (1 agent : X H_2O): Pyrol 1:10; 1:2, original (1:1=100%); TT and HTC conc. 10:1; LW 1:3.
- Conditioning, drying, volume/weight, leaching, bulking measurement following standards.
- Durability tests: Bravery Test (1978); fungi: *Trametes versicolor,* time of exposure 6 weeks.
- Durability samples: 9 samples of 5 x 10 x 30 mm^3 (Bravery 1978) for each treatment.
- Swelling measurement: 10 samples of 14 x 14 x 28 mm^3 for each treatment.
- UMSP: UV-light absorption at 278 nm, using Zeiss-UMSP 80 (Koch and Grünwald 2004).
- Light and UV-microscopic investigations.

Table 1

Chemical sources and process for impregnation of wood with solutions of native origin

No.	Used Raw-Materials	Abbreviation	Synonym	Process Conditions
1	Miscanthus sp.	HTC_Misc	HTC-AG, HTC- D. Tschok,Misc.3/2013	Hydro-Thermal-Carbonisation = HTC, pressure, temp.
2	Spruce sawdust	HTC_Saw	HTC/ S. Vondran	HTC, pressure, temp.
2.2	Brewery residuest	HTC_Brew	HTC Schlamm	HTC, pressure, temp.
3	Mixed spruce, beech, oak, ash, poplar	TT_180	Thermal Treatment, Heat treatment, HT	TT, 180°C
4	Mixed spruce, beech, oak, ash, poplar	TT_200	Thermal Treatment, Heat treatment, HT	TT, 200°C
5	Canadian beech	Pyrol_Can	Bio-Oil, BO, Old Canadian	Fast pyrolysis, Canadian process (Dynamotive)
6	European beech	Pyrol_ProF1	German 1	Slow pyrolysis, D-ProFagus process
7	European beech	Pyrol_ProF2	German 2	Slow pyrolysis, D-ProFagus process
8	European beech	Pyrol_ProF3orig	Hamburg bio oil, crude Pro Fagus	Slow pyrolysis, D-ProFagus process
9	European beech	Pyrol_ProF3low	Hamburg low fraction,CHNSCCO2	Slow pyrolysis, D-ProFagus proc., supercritical CO_2-extr. low molecular weight
10	European beech	Pyrol_ProF3big	Hamburg PH 200, residue	Slow pyrolysis, D-ProFagus proc., supercritical CO_2-extr. high molecular weight
11	European beech	Pyrol_NLfre	Btg wood oil fresh, NL fresh	Fast pyrolysis, NL- BTG process, fresh production
12	European beech	Pyrol_NLold	Btg wood oil from storage, NL old	Fast pyrolysis, NL-BTG proc., stored/ old production
13	Robinia-heartwood, milled wood	Rob_Extr		Methanol-water 1:1 extract of heartwood, 3 concentrations
14	Forest poplar	LW_Pop_for1:1	LW poplar 1:1	Liquified wood
15	Forest poplar	LW_Pop_for1:3	LW poplar 1:3	Liquified wood
16	Plantation poplar	LW_Pop_plant	LW poplar, fast growing	Liquified wood
17	Forest spruce	LW_Spruce	LW spruce	Liquified wood

RESULTS AND DISCUSSION

Chemical Analysis of Robinia Extracts and thermally produced liquid Agents

Extraction process of Robinia wood with methanol-water mixture 1:1 resulted in a wide range of different concentration levels of extractives (Fig. 1). Most substances showed maximal concentration in older Robinia heartwood compared to median or young plantation wood; Robinia sapwood and especially bark have the lowest concentration of single extracts, mainly ranked from robinetinidin > dihydrorobinetin > robinetin > gallic acid > fisetin >> catechine, whereas barks shows higher catechine amount (Figure 1; Sablík and Rademacher 2013; Sablík et al. 2016).

Chemical Analysis of Process Residues after Pyrolysis Treatment of Biomass Materials

The dominating molecules in most pyrolysis process products are gallic acid, furfural, catechol and phenol, reaching or exceeding 1000 µg per gram pyrolysis liquid (Fig. 2 left). Also 5-methylfurfural, syringaldehyde and eugenol reach in some processes a range of 200-400 µg g^{-1}, whereas all other detected molecules show concentrations <<100 µg g^{-1}. Some molecules, like gallic acid, have a widely similar range of concentrations (880-1150 µg g^{-1}), while others (div. ProFagus products; Bodenfelde/ DE; e.g. ProF3) show a 2-3 times higher amount, especially compared to NL- or CAN- products.

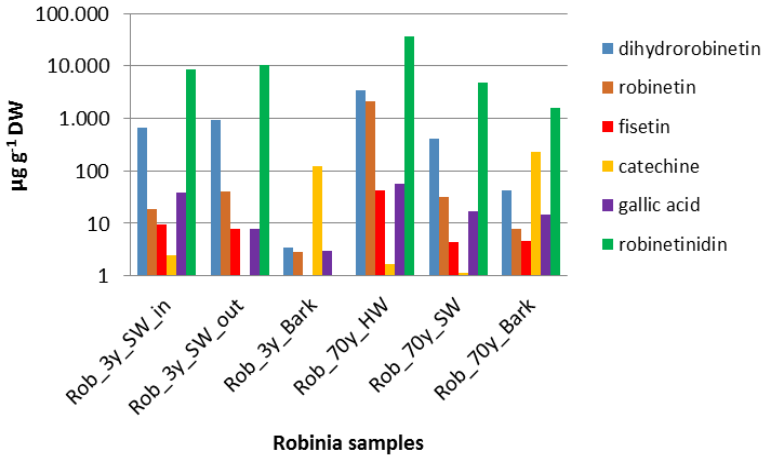

Fig. 1.

Analysis of methanol – water 1:1 extracts from Robinia (Rob): Influence of age (years; sapwood SW, heartwood HW), and stem compartment (inner and outer parts of disc, Wood or Bark) on amount of flavonoids and phenolic compounds.

Chemical Analysis of Process Residues after Thermal Treatment or Hydro-thermal-Carbonisation of Biomass Materials

In the liquid process-residues after thermal treatment of wood (TT using 180 or 200°C) or hydrothermal carbonisation of organic waste assortments (Miscanthus grass, or spruce sawdust) the robinetin-derivatives as well as fisetin, catechin and gallic acid are present in higher concentrations in the HTC-process residues (about 0.5-10 µg ml^{-1}) compared to the TT process (mainly 0.1-0.8 µg ml^{-1}, gallic acid also 1-5 µg ml^{-1}). Furfural even appears only in the HTC-Sawdust (300 µg ml^{-1}), phenol only in the TT process (30-300 µg ml^{-1}). Only acetic acid in the TT process is with up to 10 µg ml^{-1} about 10 times higher concentrated than in the HTC residues (Fig. 2 right).

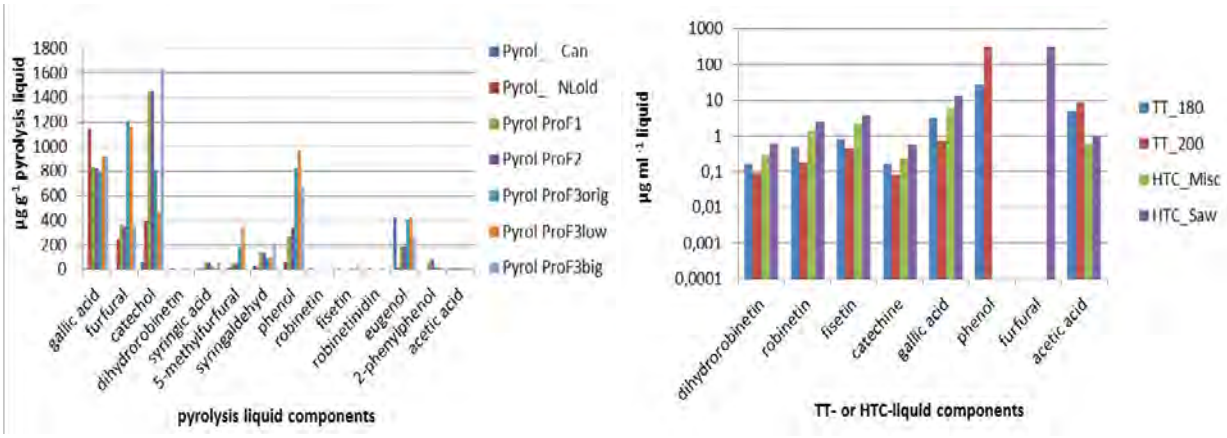

Fig. 2.

Chemical analysis of pyrolysis liquids with HPLC/ DAD in µg g^{-1} (due to high viscosity; left) and of thermal treatment residues from thermal modification with (TT: 180 and 200°C), or hydro-thermal-carbonisation (HTC: Miscanthus, spruce-sawdust) with HPLC/ MS in µg ml^{-1}.

Microscopic Observations and UV Detection of impregnated Modification Agents

Microscopy of pyrolysis-impregnated (Pyrol_Can, Fig. 3, below) beech (left) and poplar wood (right) show – compared to untreated native wood (Fig. 3, above) - a distinct deepening of the colour in the cell walls and a strong colour deepening in the cell lumina due to the more or less achieved full-impregnation of the entire micro- and macro-porous space of the wood tissue (Fig. 3). The maximal WPG, realized by full impregnation of the cell walls as well as the filling of all cell-lumina – has no further positive effect on the property improvement, because mainly the cell wall impregnation with the interaction of the impregnation agent and the cell wall hydroxyl groups results in chemical and micro-structural changes. In opposite to the cell wall impregnation the lumen filling mainly results only in an unwanted:

- Increase of the weight and colour

- Smearing of the surface and pollution of the surrounding
- Waste of treatment agents and increase of manufacturing costs

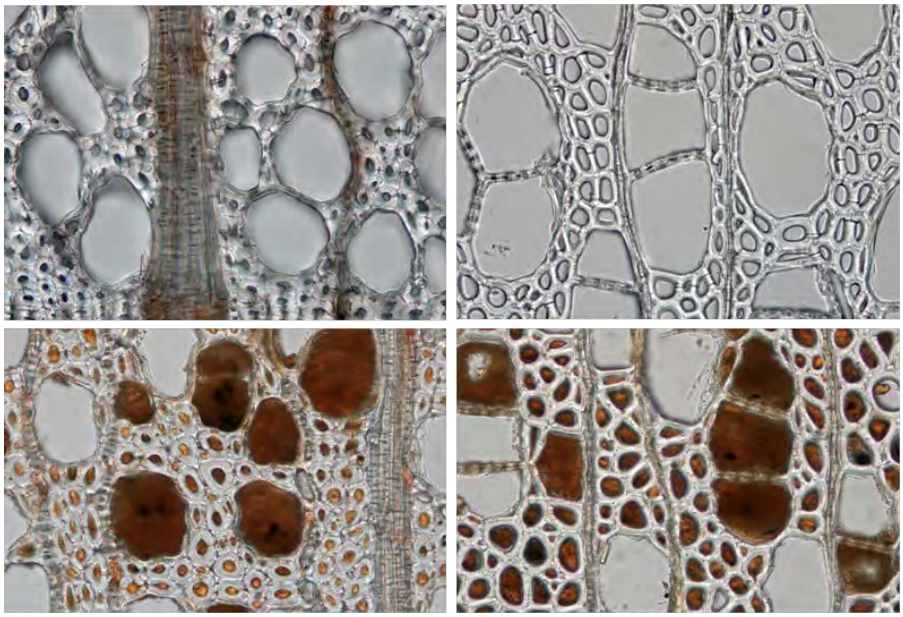

Fig. 3.
Natural (above) and pyrolysis-liquid impregnated (below) beech (left) and poplar wood (right). Photos: R. Rousek.

In order to counteract these disadvantages a post-vacuum or leaching treatment of the impregnated wood just following the impregnation process helps to minimize these problems (Fig. 4, left: after impregnation, Fig. 4, right: after leaching & post vacuum treatment) by eliminating the free liquid from the macro-pores.

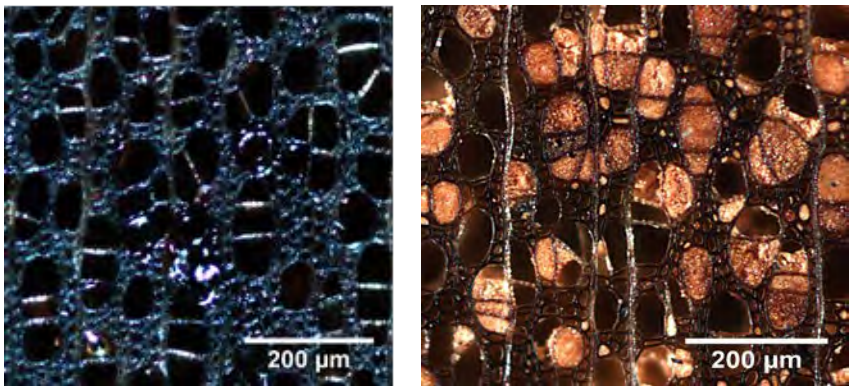

Fig. 4.
Surface images of poplar impregnated with 50% pyrolysis liquid (left; Pyrol_Can) and sample after leaching & post-vacuum (right). Photos: R. Rousek.

The microscopic view of UV-light illuminated cross sections of older Robinia heartwood as well as impregnated beech and poplar samples showed a stronger detection of phenolic compound response in kind of yellow-green fluorescence shining compared to younger Robinia or non-treated beech or poplar samples (blue colour). There was a ranking of shining, starting with native beech and poplar wood < young Robinia < leached Robinia-extract impregnated beech and poplar < Robinia-extract impregnated beech and poplar wood < old Robinia heartwood, indicating increasing phenolic compounds in the cell walls (Fig. 5).

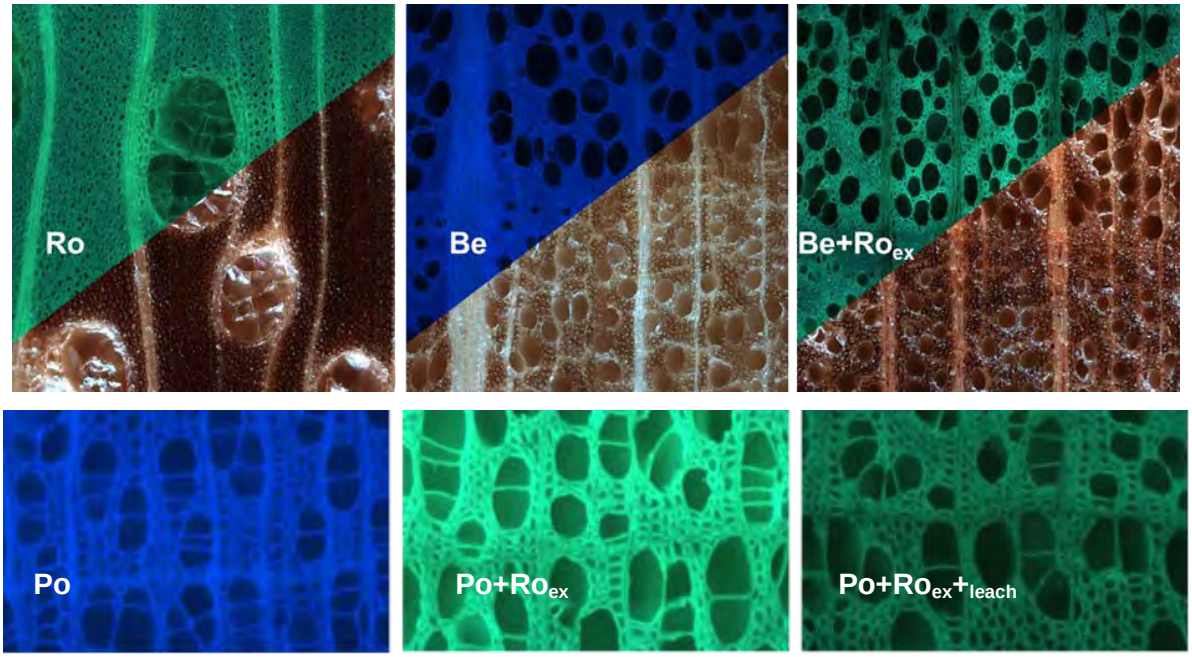

Fig. 5.
Above: Increase of UV-light induced green fluorescence shining, detecting increasing amount of phenolic compounds in cell walls of: Robinia heartwood (left; upper part of pictures: green = detection of phenolic compounds); native beech (middle; upper part of picture: blue = no phenolic compounds); Robinia - extract impregnated beech wood (right: upper part green = phenolic extract detection) compared with normal light (lower parts of upper 3 pictures: brown). Each image has a dimension of 0.9 x 0.9 mm^2.
Below: UV-light induced fluorescence shining in poplar wood: left: Reference, no impregnation (→ no green, but blue); mid: after Robinia extract impregnation (intensive green shining); right: after leaching (low green coloured). Photos: R. Rousek.

Cellular UV-Microspectrophotometry (UMSP)

UMSP images of untreated as well as pyrolysis-liquid and Robinia-extract treated poplar wood showed high differences in UV-light (278 nm) absorption, indicating higher amount of aromatic, condensed components in impregnated cell walls compared to untreated wood of native poplar (Fig. 6). The presence of these components also in leached samples confirms their strong cell wall fixation.

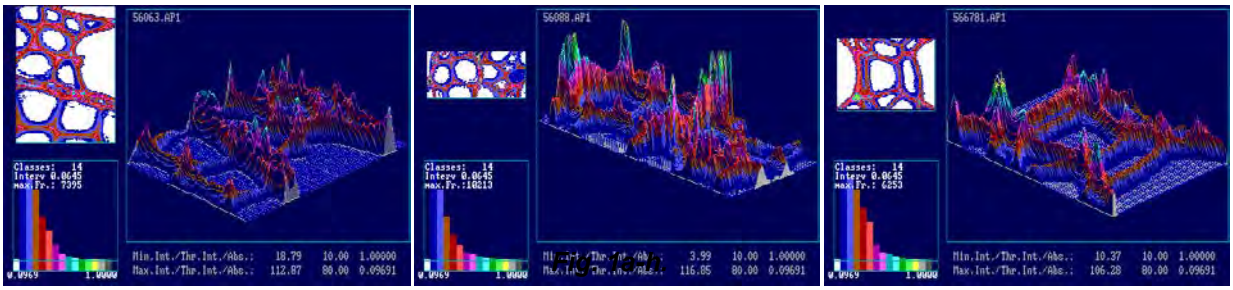

Fig. 6.
UV-absorption UMSP-field scan of reference (left) and pyrolysis-treated poplar (Pyrol_Can, mid) as well as Robinia extract impregnated poplar wood (right); wave length-setting of absorption 278 nm, absorption scale 1.0.

Weight Percentage-Gain (WPG), Weight Percentage–Remain (WPR) and Bulking Effect

The maximal WPG, possible to be reached after vacuum-pressure impregnation with thermal liquid residues is due to the lower density and higher porous system in pine sapwood about 2-times higher (up to 120% of wood dry mass) than in beech (30-70%; Fig. 7, left). In poplar wood, which has a similar or even lower density than pine, this difference as compared to beech is much lower, explained by the fact, that the facultative heartwood of poplar is classified as 'difficult to impregnate', whereas the sapwood is easily to treat like pine sapwood. (Kumar and Donriyal 1991; Hoffmann 2008;

EN 350:2016). Due to the washout of non-fixed agent (Fig. 7, mid) after leaching test only 25-40% of the impregnated pyrolysis liquids remained in the wood, in case of low-molecular fraction after supercritical CO_2-fractionation of pyrolysis liquids and in case of liquid wood from poplar and especially from spruce the washout was even higher: only 25% of liquefied poplar wood and 15% of liquefied spruce remained in the wood, in case of low-molecular pyrolysis fraction even only 10-15% of the impregnated amount remained. The corresponding WPR was 12-45% of the wood mass in case of pyrolysis liquids, 10-30% of the liquefied wood and 5% of the low molecular fraction of pyrolysis liquid (Fig. 7, mid).

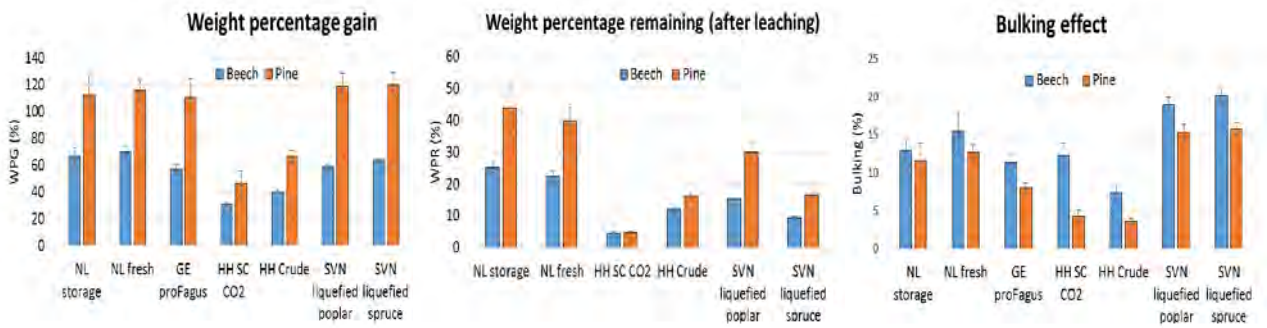

Fig. 7.
Weight-percentage-gain (left), WP-remain (right) and Bulking (permanent swelling [%; right]) without (WPG) and after leaching (WPR) of impregnated beech and pine wood using wood-processing residues (pyrolysis-liquid, liquid wood). Residues and process compare Tab. 1.

The determined bulking effect (permanent swelling of the wood due to structural modification (e.g. cross linking of hydroxyl groups in the cell wall) of the remaining agents after the leaching test was surprising (Fig. 7, right): In spite of the highest washout-rate in case of the low-molecular pyrolysis fraction and the high washout of the liquefied wood the bulking effect is still in average (low pyrolysis fraction) or even maximal (liquefied wood). This indicates that not only the amount of impregnated or remained impregnation agent is responsible for the wood-property improvement, but also the selection of reactive components with special functional groups (Ermeydan et al. 2012), especially in case of higher active slow-pyrolysed fractions after fractioning or wood liquefaction.

Moisture related Behaviour of the modified Wood

Compared to this, liquid thermal residues from thermal treatment, hydro-thermal carbonisation or Canadian fast pyrolysis process show a related behaviour between the amount of impregnated agent (WPG) and the corresponding bulking effect as well as the decreasing volume swelling response under moisture conditions (Fig. 8).

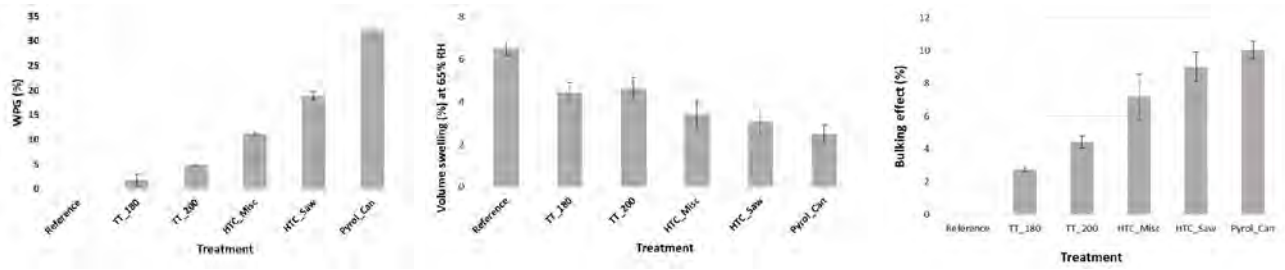

Fig. 8.
Weight percentage-gain (%; left) of impregnated poplar using wood-processing residues (thermal treatment (TT: 180 and 200°C), hydro-thermal-carbonisation (HTC: Miscanthus, spruce-sawdust) and pyrolysis-liquid from Canadian producer (Pyrol_Can), reduced volume-swelling (%; mid) and bulking effect (%; right). Residues and process compare Table 1.

The water uptake of pyrolysis and liquid wood impregnated beech wood shows (after leaching) a strong relationship (r^2 = 0.91) between the amount of impregnated agent (WPG) and the amount of included water (Fig. 9, left).

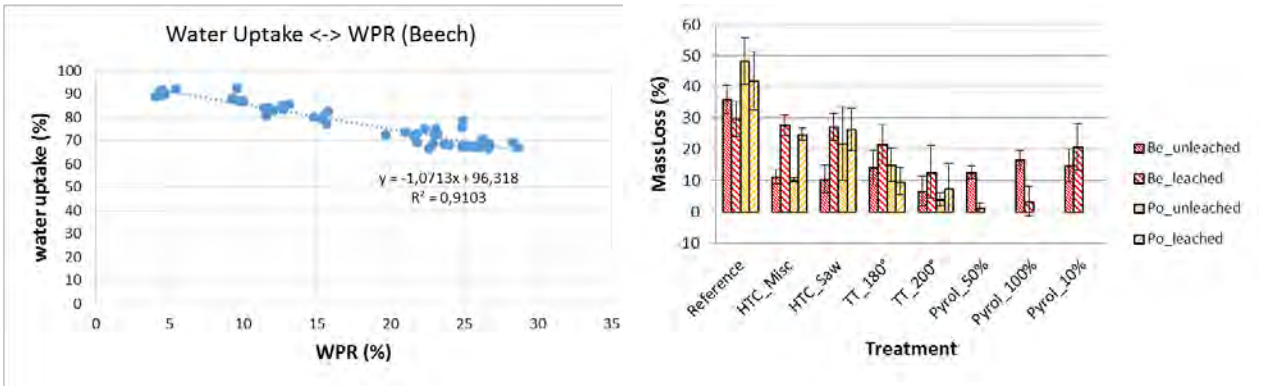

Fig. 9.
Reduced water uptake (%; left) in relation to weight-percent-remain (WPR [%]) after leaching of impregnated beech using wood-processing residues (pyrolysis-liquid, liquid wood). Mass loss of beech and poplar wood (%, right) impregnated with native residues from technical processes. Pyrolysis-Liquids (Pyrol) of different dilution degree with water, TT = Thermal-Treatment residues, 180 rsp. 200°C, HTC solution = Hydrothermal Carbonisation of Miscanthus and Spruce sawdust residues. Residues and process compare Table 1.

Fungal Decay and Durability

The mass loss due to fungal decay (*Trametes versicolor*) of native beech (30-35%) and poplar wood (40-50%) was reduced to 2% in the case of pyrolysis treatment and to 4% in the case of TT, depending on the wood species, impregnation method, concentration and leaching process (Fig. 9, right, n = 9). HTC-treatment and also TT showed acceptable results only in the case of un-leached samples, whereas leaching resulted in 10-30% decay due to fungal activity; in contrast, the pyrolysis-treatment of beech in concentration of 100% and 50% showed good durability - less than 3% of decay - also in leached samples, resulting in durability classes 2 or even 1 against basidiomycetes, whereas concentration of 10% was too low. All un-leached pyrolysis samples showed mass losses due to leaching of exceeding amount of pyrolysis liquids out of cell lumina, which was leached under culture conditions on agar and resulted in a mass loss due to lower weight after the tests.

CONCLUSIONS

The results show that an increasing demand on additional high-quality wood assortments – following rising environmentally friendly standards – can be delivered by new bio-based processes, producing and applying renewable and sustainable produced wood impregnation agents for various wood modification processes. These innovations allow that not only the material wood, but also the entire wood modification process, can operate as a really renewable and sustainable process, excluding more and more fossil sources from the material production and application processes.

Nevertheless, the industrial implementation of natural and renewable produced wood modification or impregnation agents depend on the technical and constitutionally frame conditions. Under the viewpoint of sustainability in nature and production it has to be discussed, compared, balanced, and decided, if the application of defined and tested single modification agents from fossil origin, or the use of an undefined mixture of plant components and their process residues are more confirming with the requirements of sustainability, close to nature approach, environmental and health protection as well as renewability.

ACKNOWLEDGEMENT

Support from the COST Action FP1407 ModWoodLife project, the Swedish Research Council Formas project 942-2016-64, European Social Fund and the state budget of the Czech Republic, project "The Establishment of an International Research Team for the Development of New Wood-based Materials" reg. no. CZ.1.07/2.3.00/20.0269, by the Internal Grant Agency (IGA) of Faculty of Forestry and Wood Technology, Mendel University in Brno (IGA V 79/2013), the EFOP-3.6.1-16-2016-00018 – Improving the role of research, development & innovation in the higher education through institutional developments assisting intelligent specialization in Sopron and Szombathely and European Commission for funding the InnoRenew CoE project (GA 739574) are gratefully acknowledged.

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EUROPEAN CO-OPERATION IN WOOD RESEARCH FROM NATIVE WOOD TO ENGINEERED MATERIALS, PART 2: DENSIFICATION MODIFICATION IN PRODUCT DEVELOPMENT

Peter RADEMACHER, Matyas BADER and Robert NEMETH
University of West Hungary, Bajcsy-Zs. u. 4, H-9400 Sopron, Hungary
E-mail: peter.rademacher@nyme.hu, bader.matyas@phd.uni-sopron.hu, nemeth.robert@uni-sopron.hu

Radim ROUSEK, Petr PARIL, Jan BAAR, Stanislav HORNICEK, Ales DEJMAL, Jakub DÖMENY
Mendel University in Brno, Dept. of Wood Science, Zemedelska 3, 61300 Brno, Czech Republic
E-mail: radim.rousek@gmail.com, parilpetr6@gmail.com, Jan.Baar@seznam.cz, stanislav.hornicek@mendelu.cz, ales.dejmal@gmail.com, j.domeny@seznam.cz

Jozef KÚDELA
Technical University in Zvolen, Dept. of Wood Science, T. G. Masaryka 24, SK-960 53 Zvolen Slovakia, E-mail: kudela@tuzvo.sk

Andreja KUTNAR
University of Primorska, Titov trg 4, 600 Koper, Slovenia, andreja.kutnar@upr.si, and InnoRenew CoE Livade 6, 6310 Izola, Slovenia, E-mail: andreja.kutnar@innorenew.eu

Benedikt NEYSES and Dick SANDBERG
Luleå University of Technology, Wood Science and Engineering
Forskargatan 1, 93187 Skellefteå, Sweden, E-mail: benedikt.neyses@ltu.se, dick.sandberg@ltu.se

Abstract

Wood is a renewable, biological material used in numerous applications and it is growing in importance due to sustainable development efforts. Wood also suffers from a number of disadvantages, were low hardness and abrasive resistance are characteristic for low-density species. This paper presents state of the art on different wood densification processes as one emerging process technology for increased use of low-density species. The presentation is based on work by different European research groups in wood science, collaborating in the field mainly through different COST Actions. The main principles for processes are discussed, such as bulk and surface densification, as well as methods for reducing the shape memory effect of densified wood. The main challenges for the future are in the field of finding fast and environmental friendly method for elimination of the set-recovery and scaling up to profitable industrial applications. To provide a better understanding with this regard, some relevant applications of densified wood are presented.

Key words: *compression; flooring; plasticization; thermal-hydro-mechanical processing.*

INTRODUCTION

Densification, i.e. a viscoelastic thermal transverse compression to achieve a permanent deformation of wood cells and thereby an increased in density of a piece of wood or of a part of it, is one of the approaches to improve the properties of wood that has been the subject of many studies during the recent decades (Sandberg et al. 2013). The main goal of densifying wood is to increase its hardness and surface abrasion resistance, but also in some cases to increase its strength.

For many decades, humans have been consuming more resources than the world has to offer in the long run. Increasing the use of renewable materials, such as wood, is essential if we are to achieve a sustainable use of the resources available to us. Densification has the potential to improve the properties of widely available low-density wood species, opening up new fields of application, and fostering the use of wood products in general. Figure 1 shows an example of how low-density poplar from the Central Czech Republic (clones Max 4) was used in a Czech-Slovenian-US project to improve the strength properties by heating and compression. The density was increased up to three times compared to undensified poplar with the result that modulus of elasticity (MOE) also increased considerably (Hornicek et al. 2015; Rademacher et al. 2016).

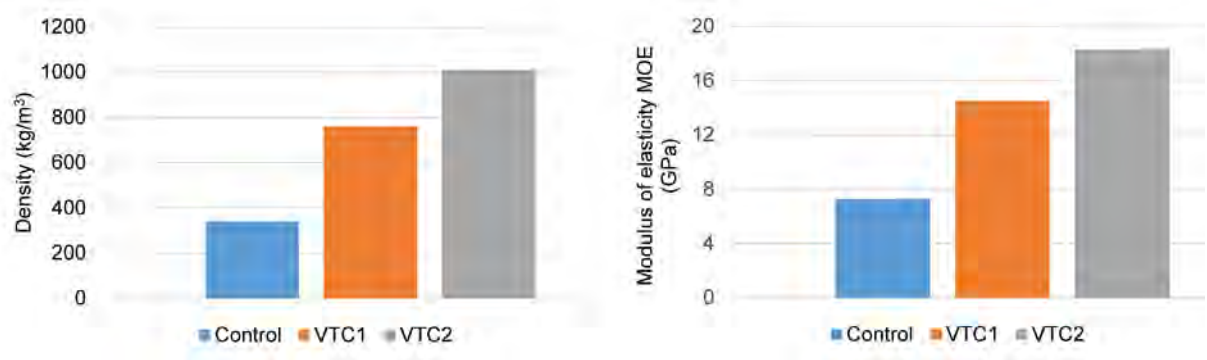

Fig. 1.
Densification of light-weight poplar wood by the VTC-process, i.e. heating and steaming in a closed-system at 170-200°C for about 7 min and compression. The compression ratio was 1:2.3 for control to VTC1 samples and 1:2.9 for the CTRL to VTC2 samples.

OBJECTIVE

The main objective of the present paper is to give a concise state-of-the-art presentation of the most recent developments in the field of densification of wood in product development.

THE DENSIFICATION PROCESS

The degree of densification of a piece of wood can be varied depending on what it is desired to achieved. To increase the gross density and thereby improve the overall mechanical properties of the wood material, all the cells throughout the thickness must be compressed and deformed (through-thickness densification). The gross density can theoretically be increased to a value close to that of the cell wall, i.e. 1500 kg/m^3. The energy consumption for this densification is very high, especially in the final phase of compression when the denser cells in the annual rings are to be deformed.

If the purpose is just to achieve a hard and abrasive-resistant surface, only the wood cells close to the surface need to be compressed and deformed (surface densification). Compared to through-thickness densification, surface densification offers several advantages. From a structural perspective, surface-densified wood has a higher material usage efficiency – imagine an I-beam versus a solid rectangular beam. For some products, better dampening characteristics are an asset. In addition, the compression and treatment to avoid the moisture-induced recovery of the densified wood cells back to their original shape need only affect the densified cells close to the surface and not the whole piece of wood. This may allow a faster, less energy-consuming, and thereby a less costly treatment process.

Regardless of how the densification itself is performed, the process basically always consists of three main phases: (1) softening of the wood material to be densified, (2) densification of the cells, and (3) elimination of the set-recovery. These phases interact with each other, but are here for simplicity described as separate processes. Other methods such as a modified wood welding technique (Vasiri et al. 2014; 2015) or self-bonding compression techniques (Cristescu et al. 2015a,b) can, however, be used for surface densification, but is not discussed further here.

Softening

Wood is a material that can be made plastic (or semi-plastic because it has areas with a crystalline molecular structure that are hard to plasticize), which means that wood can be softened and shaped so that it keeps its new shape after the plasticization is finished. The observed glass transition of wood occurs over a temperature range of ca. 50°C, and it is dependent on the moisture content and the time domain of the observed mechanical response. An increase in temperature or moisture content decreases the compressive modulus of wood. A combination of moisture and heat (hydro-thermal treatment) is therefore an effective way of softening wood, but chemical methods can also be used with excellent results, cf. Fig. 2. Nilsson et al. (2011) described a simple densification technique based on compressing Scots pine sawn timber in the radial direction, which shows no or little cell-wall fracture even if the densification is performed at 20°C at a moisture content of about 8%, i.e. un-plasticized. The densified wood was used as the surface layer of a multi-layer composite with a light-weight core. In the through-thickness densification of wood, however, plasticization treatment is an extremely important part of the process to soften the wood sufficiently in the specific region were densification is desired, and so that can without fracture withstand the compressive deformation.

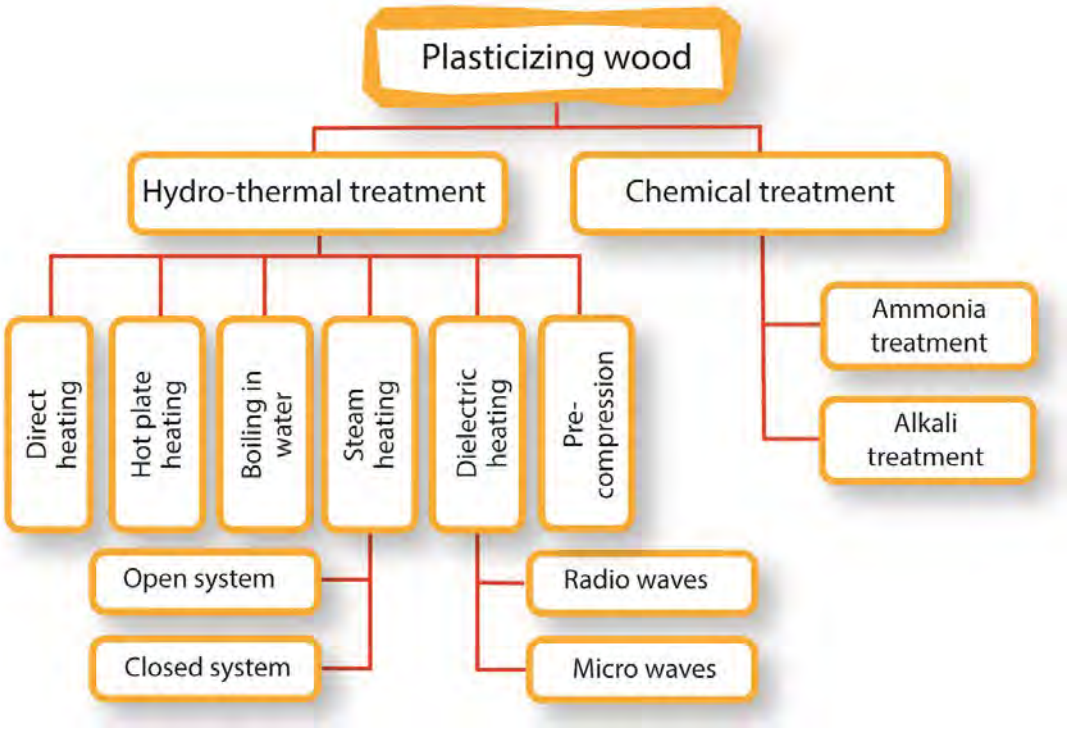

Fig. 2.
Classification of methods for making wood more flexible for shaping (Navi and Sandberg 2012).

Chemical treatment for plasticization can be as effective as hydro-thermal processes (Stojčev 1979). In one study (Rousek et al. 2015), plasticization by vacuum impregnation (0.2 MPa) with ammonia gas at room temperature was compared to plasticization treatment with saturated steam at atmospheric pressure and a temperature of 100°C. Ammonia plasticization reduced the compression forces significantly and was sufficient for densification, but was slightly less efficient than saturated steam, Fig. 3. The ammonia plasticization effect could be improved by applying a higher gas pressure. The best plasticization effect was reached by combining these two methods.

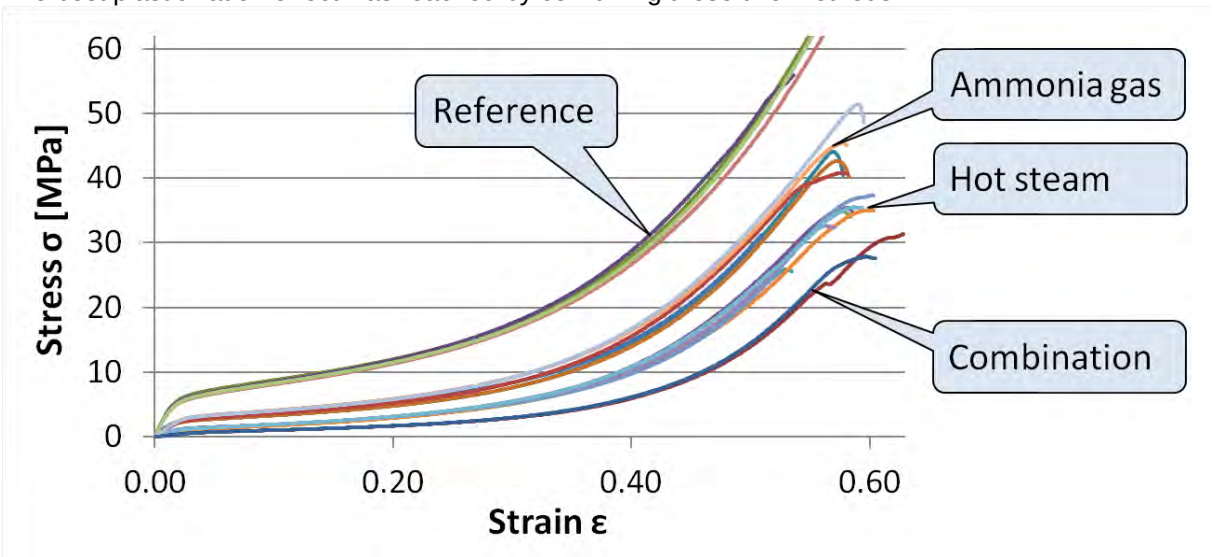

Fig. 3.
Stress-strain relationship for tangential compression of untreated beech (reference) and beech softened by ammonia gas, saturated steam or both in combination (Rousek et al. 2015).

Microwave heating is a time-reducing method of plasticization that has been studied by e.g. Norimoto and Gril (1989) and Dömény *et al.* (2014; 2017). Plasticization by MW heating has several advantages over the conventional water vapour method, such as lower energy consumption, rapid

heating of wood over the whole volume and application in continuous processes. In contrast, structural changes can occur during MW treatment and this may lead to changes in wood strength (Oloyed and Groombridge 2000). When microwave radiation is applied to the wood with a certain moisture content (MC), the water in the cell cavity absorbs the energy and is vaporized. The high internal steam pressure can cause the cell to rupture and generate micro cracks in the wood structure.

Densification

Densification can be achieved in one or more directions, but the process is mostly performed along one of the orthotropic axes, and the efforts are more successful for diffuse porous hardwoods than for softwoods. Densification can optimally be achieved mainly in the radial direction for softwoods and in the tangential direction for hardwoods with large aggregated rays. If e.g. Scots pine is densified along the tangential direction, the latewood (LW) of the annual rings spreads into the earlywood and forms waves or a zigzag pattern and the whole piece of wood may be crushed (Sandberg 1998a,b), Fig. 4. Densification in the radial direction, on the other hand, flattens the cells without any noticeable damage at the micro- or macroscopic level. In hardwoods with large aggregated rays, the same type of crushing phenomenon happens with the rays, Fig. 4 (Rousek et al. 2015).

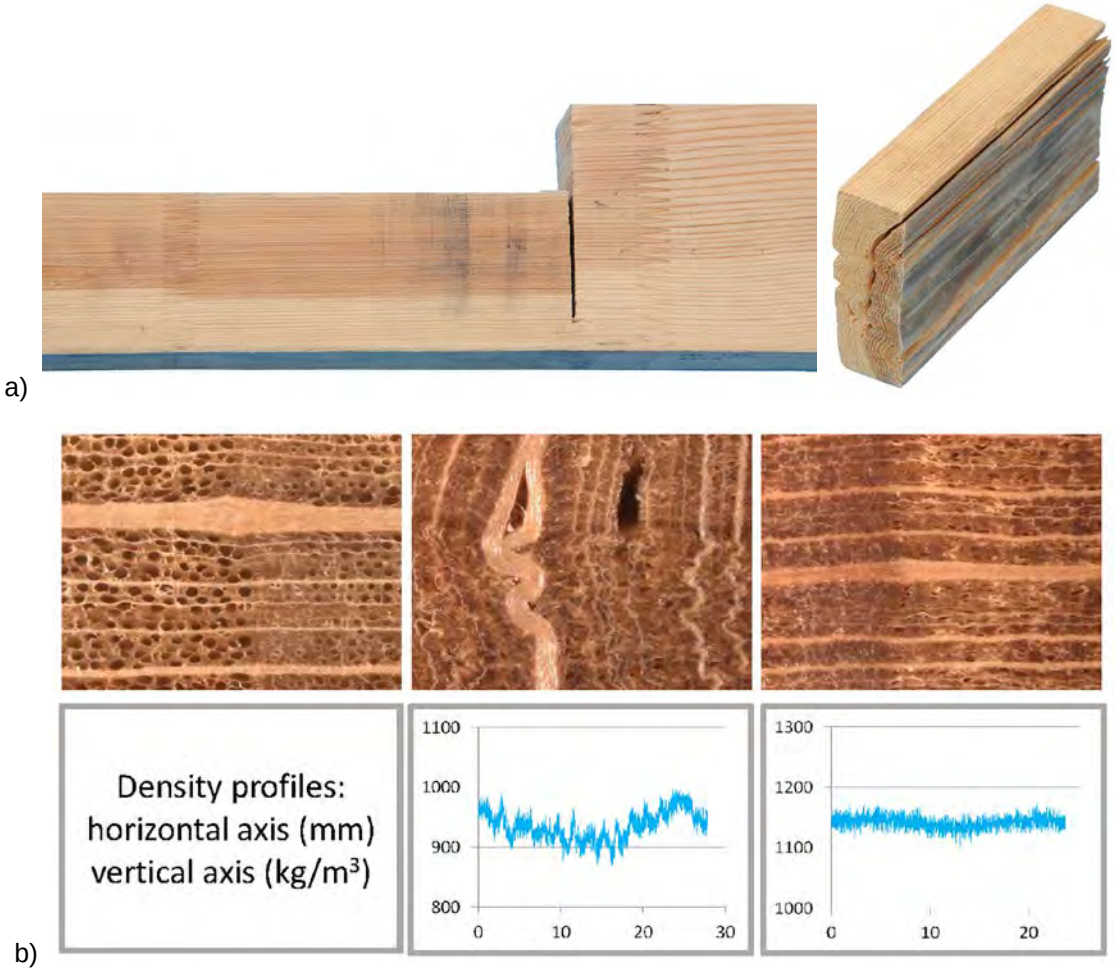

Fig. 4.
Densification of wood: a) Macro-views of densified Scots pine; densification in radial direction of a finger-jointed 50x100 mm board (left) and damage in a tangentially densified 22x75 mm board (right). b) Cross-section microscopic views of un-densified beech (left), and of beech densified in the radial (middle) and tangential (right) directions. The diagrams show the density profile through the cross-section after densification.

During transverse compressive loading, the typical stress-strain curve of wood has three distinct regions corresponding to three different types of cell deformation: 1) A linear elastic part, 2) a 'collapse' region where the stress is almost constant even at high strain, 3) a sharp increase in stresses due to contact between the inner cell walls (cf. Fig. 3). Cellular collapse occurs by elastic buckling, plastic yielding or brittle crushing, depending on the test conditions and on the nature of the

cell wall. It is also known that wood responds differently to radial and tangential compression. Rousek (2014) has described how the strain varies within beech under densification according to the annual ring orientation in its cross-section, Fig. 5.

In radial compression, earlywood primarily controls the elastic and plastic parts of the stress-strain response, while the final compaction stage is dominated by the elastic deformation of latewood. In the tangential direction, the final compression stage begins after buckling of the latewood layers. The compressibility of different wood tissues affects the distribution of void areas, and thus also the density distribution and mechanical properties of compressed wood (Kamke and Casey 1988). The location of the buckling of both the cell walls and the annual rings during transverse densification cannot be predicted precisely, as it depends on the morphology of the microstructure and degree of plasticisation.

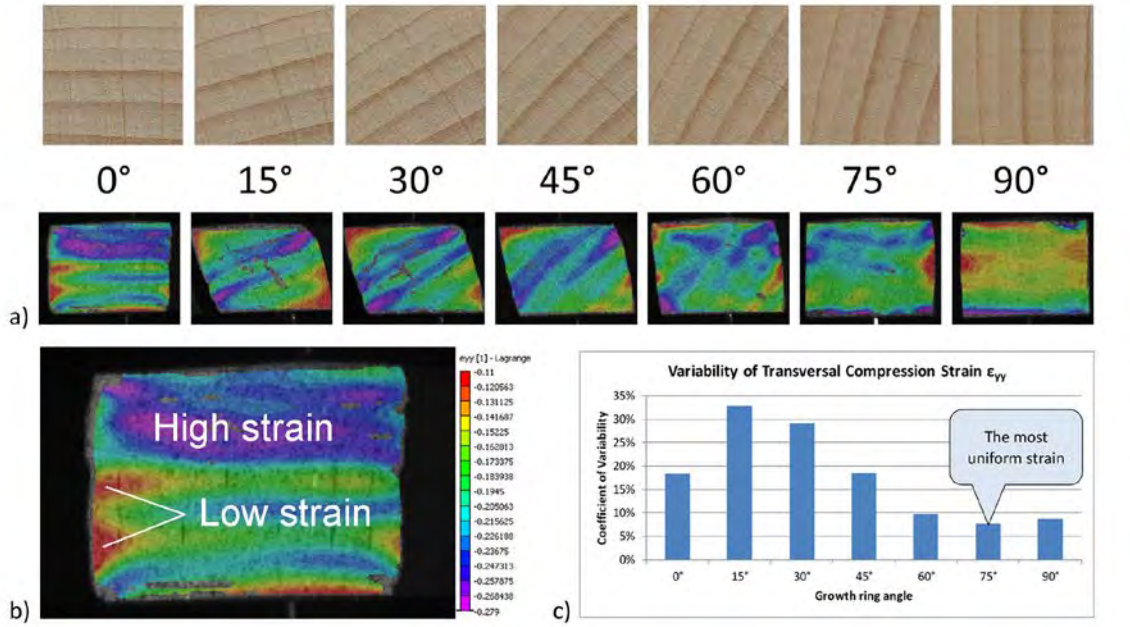

Fig. 5.
Densification of beech with different annual ring orientations in the cross section (upper): a) Digital Image Correlation (DIC) showing the strain at 26% compression, b) DIC showing the vertical strain ε_{yy} distribution in the radial compressed sample (0°), and c) the variability of the vertical strain (Lagrange) calculation Set Recovery.

Already after early studies in this field, it was clear that the unmodified and densified wood cells would recover once they were exposed to moisture. This phenomenon is related to the shape memory effect of wood called "set-recovery" and is related to the release of internal stresses within the densified wood cells. In contrast to the slow set-recovery, elastic spring-back occurs immediately when the pressure is released after the densification process. In general, the elastic spring-back can be attributed to elastic strains within the chemical bonds of the wood material. Fig. 6 shows the different stages of the densification process on the cell level and when elastic spring-back and set-recovery occur (Neyses 2016).

Five general approaches to achieve a long-term fixation of the densified wood cells have been identified: (1) mechanical fixation by gluing or impregnation with adhesives, such as epoxy resin, or nailing, screwing, etc., (2) the formation of cross-links between molecules of the wood matrix by chemical modification: deactivation of the OH-sites, for example by BELMADUR process with DMDHEU, or (3) formaldehydation (fixing of H_2CO between two hydroxyls to obtain a strong chemical bond), (4) relaxation of internal stresses within the wood matrix during densification, for example by thermo-hydro-mechanical treatment, (5) reducing the accessibility of the cell wall to water.

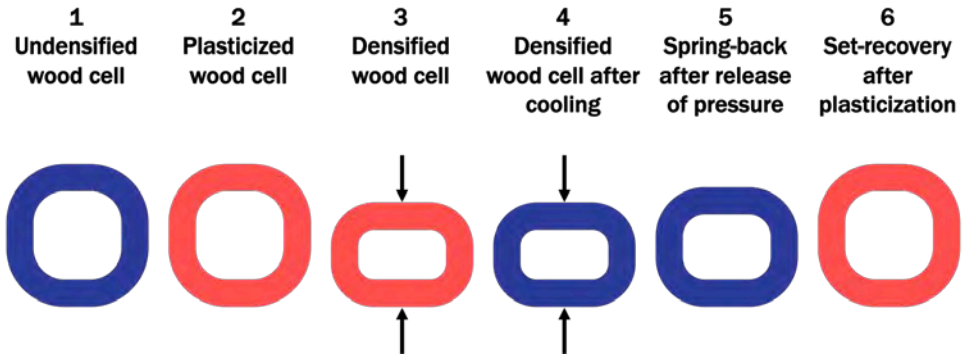

Fig. 6.
Cross-sectional view of a wood cell showing the nature of set-recovery and spring-back.

CASE STUDIES

Only a few wood densification applications have been industrialized. There are many reasons for this relatively low transfer of research to a fully up-scaled industrial production. Some of them are related to unsolved problems at the laboratory level on small-sized samples and others are related to the scaling-up processes in industry and the market. One particular reason was the lack of adequate consideration of the plasticization or stability of the products. The latter issue was solved by the development of resin-impregnated laminated and densified products, which are today being commercially produced, such as Delignit®, Dehonit®, Permawood® (also known as Lignostone®), Permali®, Insulam®, and Ranprex® (Kutnar et al. 2015).

Lignamon® is ammonified and densified beech wood. The treatment of beech by gasification with ammonia and simultaneous steaming plastifies the wood and allows densification of the material up to a density of about 1100 kg/m^3 with an appropriate increase in the mechanical properties, Fig. 7 (Pařil et al. 2014).

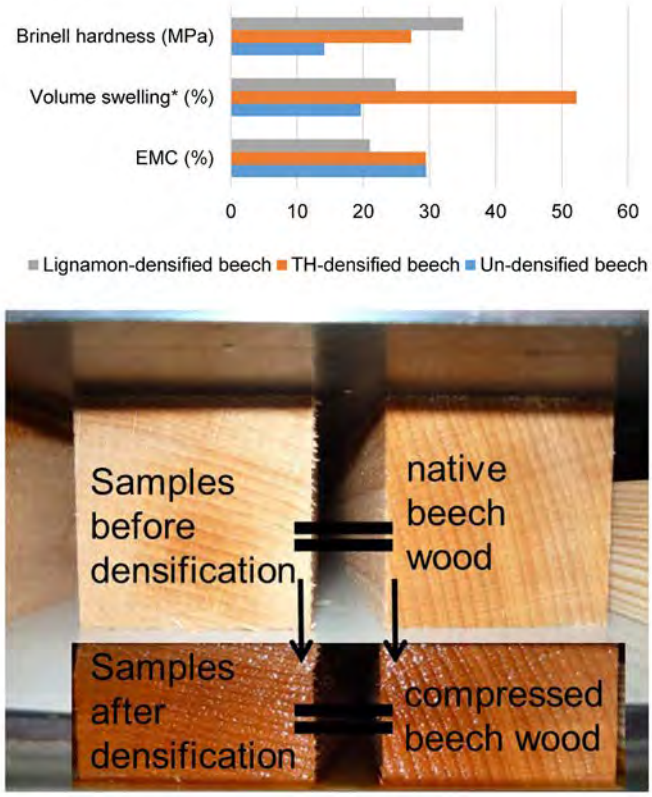

Fig. 7.
The Lignamon process delivered material with improved properties (upper) compared to control and steamed-densified beech (lower), Photo: Martin Brabec.

The production process combines vacuum-pressure impregnation of beech with ammonia vapour at a temperature of 90°C and densification. The process continues with drying, stabilization (180°C) and acclimatization (Stojčev 1979). The heating also increases the dimensional stability of the wood under moist conditions and can improve the durability compared with that of untreated beech (DC 5) up to durability class DC 1. Lignamon was presented as a material with considerably improved properties, but despite this the factory closed and Lignamon is no longer being produced.

Densification throughout the cross section may be a drawback in situations where, for example, it is desirable to maintain the low bulk density of wood, e.g. flooring applications. In such situations, an alternative approach is to densify only the first few millimetres beneath the surface of the wood, which places a great demand on the plasticisation and compression process stages to achieve the desired density profile within the wood. In one study, a high-speed continuous surface densification process was introduced, where the surface of solid Scots pine boards could be densified at speeds of up to 80 m/min by a roller pressing technique (Neyses et al. 2015). The present focus is to make the process more industrially adapted by integrating the roller pressing technique with various pre- and post-treatment methods to reduce negative effects such as set-recovery, colouring, or embossment of the surface, Fig. 8. A continuous process with steel belts instead of rollers has been studied by Sadatnezhad et al. (2017).

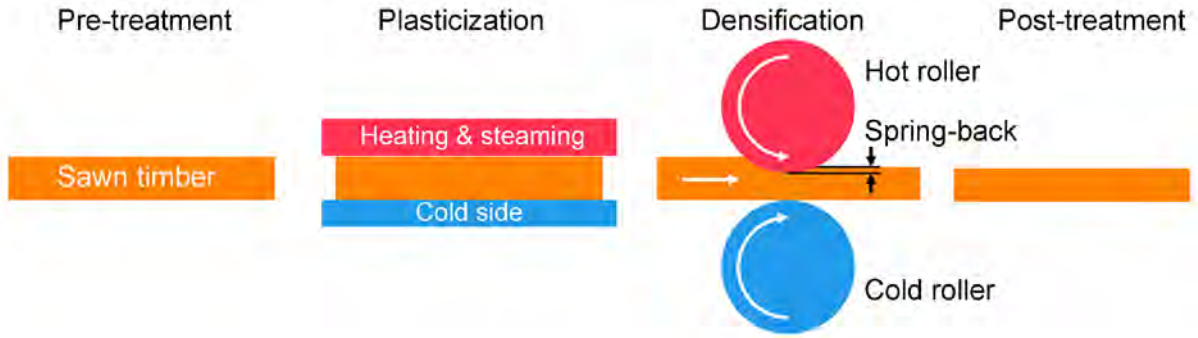

Fig. 8.
Schematic illustration of a continuous surface densification process.

Dömeny et al. (2017) used microwaves for plasticisation before and stabilization after the densification of beech. The densified beech was used as surface layer for a two-layered flooring panel where one of the layers was of densified wood and the other was particleboard, Fig 9.

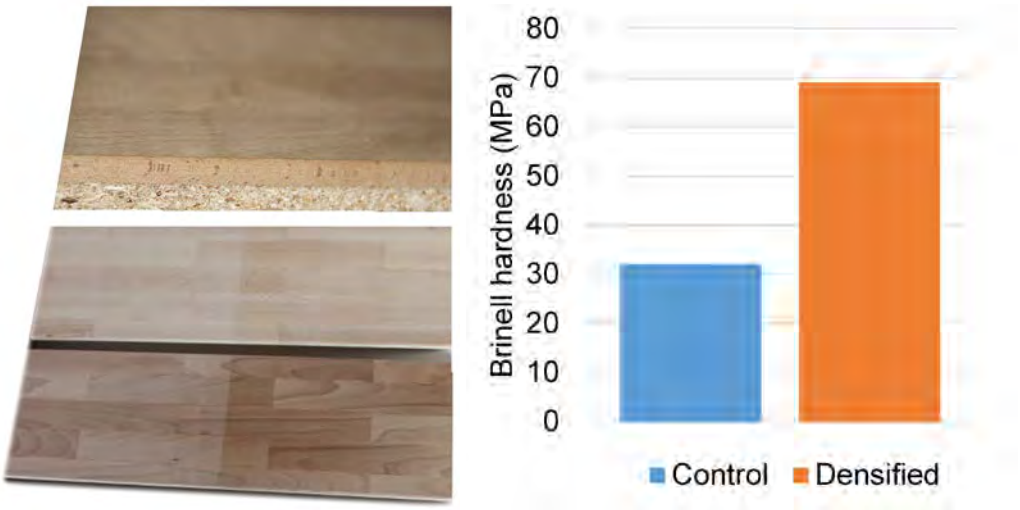

Fig. 9.
A layered flooring panel (left top) with densified beech as top surface (left bottom). The Brinell hardness value (right) of the densified beech is more than double that of the un-densified beech (left middle).

A densification process to manufacture wooden tubes out of sawn timber for load-bearing and conveying applications such as wind turbines has been presented by Kutnar et al. (2015), (Fig. 10). The tubes can be fibre reinforced at the outer face to increase strength and protect the structure. Haller et al. (2013) also investigated the use of moulded tubes in aggressive environments. Hot, highly concentrated brine was conveyed at temperatures of up to 60°C. Compared to steel tubes, the spruce tubes did not show any noticeable erosion after 4 weeks' exposure, whereas the steel tubes had been considerably affected.

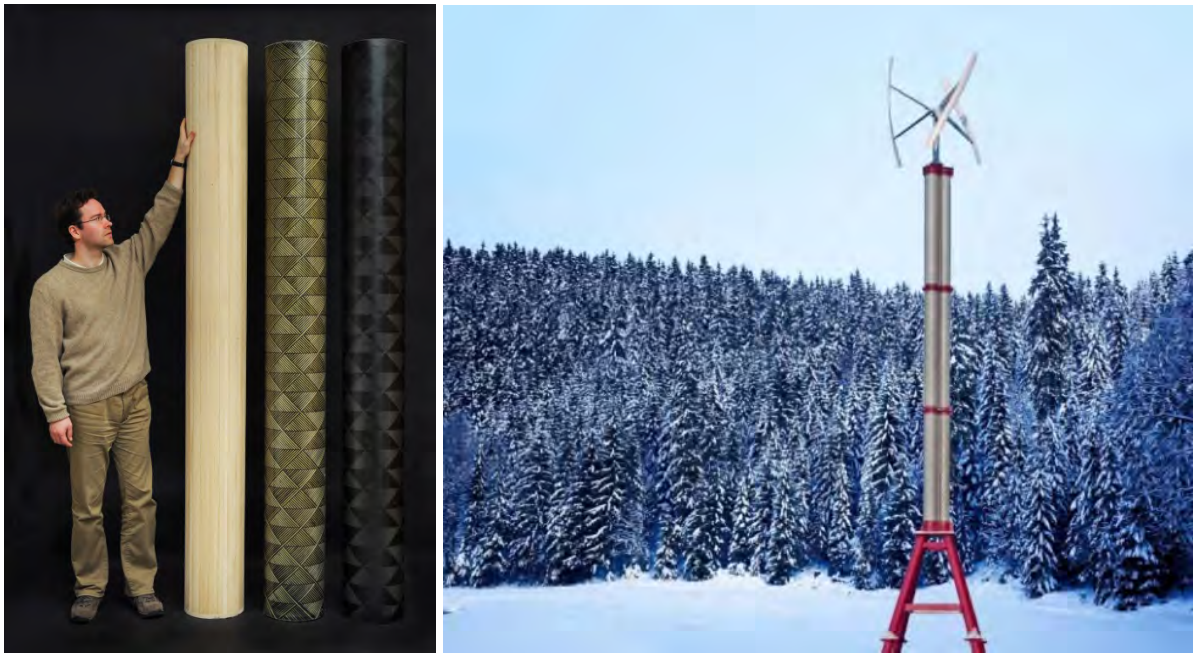

Fig. 10.
Moulded tubes from Norway spruce (left) with a length of 250 cm, a diameter of 28 cm, and a tube-wall thickness of 2 cm, and to the right a wind-power plant from fibre-reinforced moulded tube (Kutnar et al. 2015).

CONCLUSIONS

Research on wood densification is intensive in several research groups in Europe trying to understand the plasticization and densification of wood, and to develop processes and products that are environment-friendly. The focus has changed from through-thickness densification towards the densification of specific regions where the increase in density is needed in a specific product, e.g. surface densification for flooring. The main challenges for the future are finding a fast and environment-friendly method for the elimination of the set-recovery and scaling up to profitable industrial applications.

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MICROSCOPIC AND MACROSCOPIC SWELLING AND DIMENSIONAL STABILITY OF BEECH WOOD IMPREGNATED WITH PHENOL-FORMALDEHYDE

Tom FRANKE
Eberswalde University for Sustainable Development, Schicklerstr. 5, 16225 Eberswalde, Germany
Email: tom.franke@hnee.de

Axel MUND
Eberswalde University for Sustainable Development, Schicklerstr. 5, 16225 Eberswalde, Germany
Email: axel.mund@hnee.de

Claudia LENZ
Eberswalde University for Sustainable Development, Schicklerstr. 5, 16225 Eberswalde, Germany
Email: claudia.lenz@hnee.de

Nadine HEROLD
Eberswalde University for Sustainable Development, Schicklerstr. 5, 16225 Eberswalde, Germany
Email: nadine.herold@hnee.de

Alexander PFRIEM
Eberswalde University for Sustainable Development, Schicklerstr. 5, 16225 Eberswalde, Germany
Email: alexander.pfriem@hnee.de

Abstract

Three phenol-formaldehydes with varying molecular weight are investigated according to their suitability to improve the dimensional stability of beech wood (Fagus sylvatica L.*). Focus of the work is to investigate the relation of the bulking effect of the wood substance caused by the varying phenol-formaldehydes treatments to the corresponding dimensional stability on macroscopic level of small wood blocks compared to microscopic level of single cell walls. Therefore, beech blocks are treated with the certain phenol-formaldehydes, bulking is determined and subsequently the wood is exposed to three soaking-drying cycles. The evaluation was performed according to the anti swelling efficiency and leaching of the phenol-formaldehydes. On microscopic level, changes of the transverse area of the cell walls due to phenol-formaldehyde treatment and subsequent water soaking is estimated. The obtained results indicate, that penetration into the cell walls are the decisive factor for dimensional stability of the treated wood. Phenol-formaldehyde residuals in the cell lumina contribute negligibly to the dimensional stability*.

Key words: *wood modification; phenol-formaldehyde; dimensional stability*.

INTRODUCTION

Phenol-formaldehyde resins (PF) are widely used in wood working industries due to their beneficial characteristics, e.g. the hydrophobicity and weather resistance (Sonntag und Norton 1935, Hill 2006). Impregnated into wood, PF improve the wood dimensional stability and reduce water absorption (Stamm and Seborg 1960, Gabrielli and Kamke 2010).

The primary goal of the present work is the impregnation of beech veneer (*Fagus sylvatica* L.) with PF resols in order to plasticize the treated veneers and enhancing the mouldability. Furthermore, a simultaneous temperature-induced curing is suggested to improve the form stability of the modified veneers compared to the traditionally used steaming or boiling of wood. The concept of cell wall swelling by PF impregnation enabling an improved mouldability has been already evidenced (Franke 2017). An easy penetration into wood cell walls was found for low molecular weight PF (L-PF) leading to an increased plasticization in contrast to high molecular weight PF (H-PF). These results are in accordance with observations from Furuno et al. (2004) and Kajita (1991). For completion, the presents study focuses on the form stability of PF modified wood samples when exposed to water. In compliance with earlier studies and in accordance with Buchelt et al. (2012), measurements were conducted on cell wall level using microtome slices as well as on macroscopic level using small wood blocks made from beech (*Fagus sylvatica* L.). Again, three different PF resols were selected for this study representing a low (L-PF) a medium (M-PF) and a high molecular weight PF (H-PF). In order to

achieve a better understanding of the mechanisms of improved dimensional stability of PF treated wood, the differences between swelling after impregnation, bulking, and dimensional stability after three soaking-drying cyclic tests were investigated and compared to the behaviour of treated single cell walls.

MATERIAL AND METHODS

Phenolic Resins

The used water-soluble PF are products from Prefere Resins®, Erkner, Germany. Three various PF were selected for the investigations. The main characteristic feature of a PF is the varying molecular weight (Mn) and viscosity. Thereby a low molecular weight PF (L-PF), a medium molecular weight (M-PF) and a high molecular weight phenolic resin (H-PF) were investigated.

Table 1

Technical specification of the applied phenolic resins

PF type	PF concentration [%]	Molecular weight Mn	Viscosity [mPas]
L-PF	45	250	13
M-PF	55	450	196
H-PF	45	890	242

Microscopic Investigations

The microscopic investigations were performed with a light microscope (Olympus BX 41 equipped with a digital CCD-camera).

In order to compare microscopic swelling and dimensional stability of the 3 various PF, microscopic swelling was determined at 4 various states of the cell wall. Therefore, specimens were prepared from beech (*Fagus sylvatica* L.) with a transverse section of 10mm x 5mm. In a first step, transversal slices with a thickness of 15 ... 20µm were prepared and subsequently dried at 80°C to constant weight. Afterwards, 30 - 40 cell wall dimensions were measured along the middle lamella and the cell wall lumina and the cell wall area was calculated by means of subtracting the outer area minus the inner area. In the next step, same specimens were saturated with deionised water and cell wall area was determined as described before for the same cells. In the third step specimens were impregnated under vacuum conditions (100mbar for 1h and subsequently soaked for further 24h under room conditions) with the specific PF and subsequently cured at 150°C for 90min. In the last step, the cured specimens were saturated again with deionised water and cell wall areas were determined.

The area bulking coefficient (ABC) according to Buchelt et al. (2012) was determined according to equation (1).

$$ABC = \frac{Area_1 - Area_0}{Area_0} \cdot 100\,[\%] \quad (1)$$

Where ABC is the area bulking coefficient [%], $Area_1$ is the area of the treated specimen [µm²] and $Area_0$ is the area of the absolute dry state before treatment [µm²]

Dimensional stability was expressed throughout the anti swelling efficiency (ASE) in equation (3) based on the swelling coefficient (SC) displayed in equation (2) according to Rowell and Ellis (1978).

$$SC = \frac{Area_1 - Area_0}{Area_0} \cdot 100\,[\%] \quad (2)$$

where: SC is the swelling coefficient [%], $Area_1$ is the area of the treated and water soaked cell wall [µm²], $Area_0$ is the area of the absolute dry state after treatment and curing of the PF [µm²]. Respectively, SC was also determined for the untreated control.

$$ASE = \frac{SC_{ut} - SC_t}{SC_{ut}} \cdot 100 \ [\%] \quad (3)$$

where: ASE is the anti swelling efficiency [%], SC_{ut} is the swelling coefficient of the untreated and water soaked cell wall [µm²], SC_t is the swelling coefficient of the treated and water soaked cell wall [µm²].

Macroscopic investigations

For the macroscopic investigation beech blocks (*Fagus sylvatica* L.) with dimensions of 25mm × 10mm × 30mm (radial × tangential × longitudinal) were prepared. Additionally to the origin PF concentration, PF were diluted in deionised water to a PF concentration of 20%. 12 specimens for each PF (L-PF, M-PF and H-PF) and PF concentration as well as 12 untreated control specimens were used.

Prior impregnation, all specimens were kiln-dried to constant weight at 80°C. Subsequently, dimensions were determined at 2 measurement points in radial and 2 measurement points in tangential direction. Afterwards, specimens were impregnated with the respective PF and PF concentration and respectively with deionised water for the control group. Therefore, a vacuum of -85kPa was applied for 30min and subsequently a pressure of 500kPa was applied for 90min. In order to realize a maximum penetration of the PF into the wood substance, specimens were kept submerged in the impregnation solution for 24h. Afterwards the specimens were cured first at a temperature of 80°C for 90min and subsequently for 60min at 140°C. Dimensions were measured again and the WPG was calculated according to equation (4). Furthermore, the area bulking coefficent (ABC) was detemined as shown in equation (1). Hereby, radial × tangential dimensions [mm²] were used instead of the cell wall area.

$$WPG = \frac{m_1 - m_0}{m_0} \cdot 100 \ [\%] \quad (4)$$

where: WPG is the weight percent gain [%], m_1 is the weight after curing [g] and m_0 is the absolute dry weight of the untreated specimen [g].

In sum, 3 cycles of soaking and drying were carried out to determine the dimensional stability and leaching of the certain PF and their concentrations. During soaking the specimens were impregnated for 20 min with water at a vacuum of 100mbar and afterwards kept submerged for 3 days in the water. It should be noted that no vacuum was applied for the first soaking cycle. After soaking, the specimens were dried for 3 days under laboratory conditions and finally dried in a kiln at 80°C until weight constancy. After each soaking-drying cycle, the macroscopic ASE was calculated based on the SC following equation (2) and (3) (based on the radial × tangential changes of swelling in mm²) and the loss of PF during the leaching cycles expressed as leaching coefficient (LC) was determined according to equation (5).

$$LC = \frac{WPG_1 - WPG_0}{WPG_0} \cdot 100 \ [\%] \quad (5)$$

Where LC is the leaching coefficient [%], WPG_1 is the absolute dry WPG after the current leaching cycle [%] (tangential × radial) and WPG_0 is the absolute dry WPG of the current leaching cycle of the specimens [%].

RESULTS AND DISCUSSION

Beech wood treated with 20 %L-PF and M-PF display similar WPG after curing (Tab. 2), whereas the H-PF displays lower WPG values at 20% PF concentration. It is conceivable that H-PF penetration into the wood substance is retarded due to higher molecular weight and thus a higher viscosity. After exposure to 3 leaching-drying cycles, a great amount of the impregnated H-PF has been washed out. LC display values of 39.6% (45% PF concentration), respectively 27.2% (20% PF concentration). In contrast, L-PF (4.6 % at 45 % PF concentration; 8.4% at 20% PF concentration) and the M-PF (5.8% at 45% PF concentration; 4.2% at 20% PF concentration) display only a low decrease of the LC. Franke et al. (2017) observed a similar leaching behaviour of these PFs during an artificial

weathering test. Studies from Furuno et al. (2004) claims, that lower molecular weight fractions of PF can penetrate the wood cell wall, whereas fractions of higher molecular weight remain in the cell lumina. Similar claims are found by Wallström and Lindberg (1999) for pine (*Pinus sylvestris* L.) treated with polyethylene glycol. Censequently, PF in the cell lumina is leached out more easily, whereas PF in the cell walls remains stable. Hill (2006) reviewed that PF molecules form three-dimensional networks within the cell walls and thus, resist leaching. Additionally, Yelle and Ralph (2016) claimed covalent bondings between lignin and PF. This can also contribute to the stability of PF polymer inside the cell walls.

Considering the penetration into the cell wall, Fig. 1 displays the bulking of the wood blocks (at various PF concentrations) and the cell walls (PF concentration: L-PF = 45%, M-PF = 55%, H-PF = 45%) by means of the ABC. In general, the cell walls show a higher degree of swelling compared to macroscopic swelling of the wood blocks. Furthermore, it is shown that even the H-PF can induce a bulking effect to the wood cell walls, indicating a penetration into the cell walls. However, the macroscopic ABC values of the H-PF treated wood are lower than those of the L-PF and the M-PF. L-PF affects highest swelling of the material. The slightly higher concentration of the M-PF impregnation solution (55%) results in a higher WPG but lower ABC values. These results are in accordance with claims from Furuno et al. (2004) suggesting that only smaller fractions of PF penetrate the cell walls of wood, whereas greater molecular fractions remain in the cell lumina. A comparison of microscopic ABC to macroscopic ABC values demonstrate a cell wall swelling being at least twice as high as the macroscopic swelling at similar PF concentrations of L-PF and M-PF. In contrast, the H-PF displays different behaviour. Macroscopic swelling at 45% PF concentration is 1/6 lower than the swelling of the cell wall. AS previously assumed, the penetration of H-PF through the wood substance might be limited due to the high molecular size and viscosity. At 20% PF concentration L-PF and M-PF show a reduction of the ABC, but still swelling values around 10%. In contrast, the macroscopic ABC for the H-PF at 20% PF concentration displays negative BC values. resulting from poor penetration of the cell walls and thus, a collapse of the wood substance due to stress during curing of the PF at 140°C. A similar material behaviour of wood impregnated with H-PF was also determined by Furuno et al. (2004).

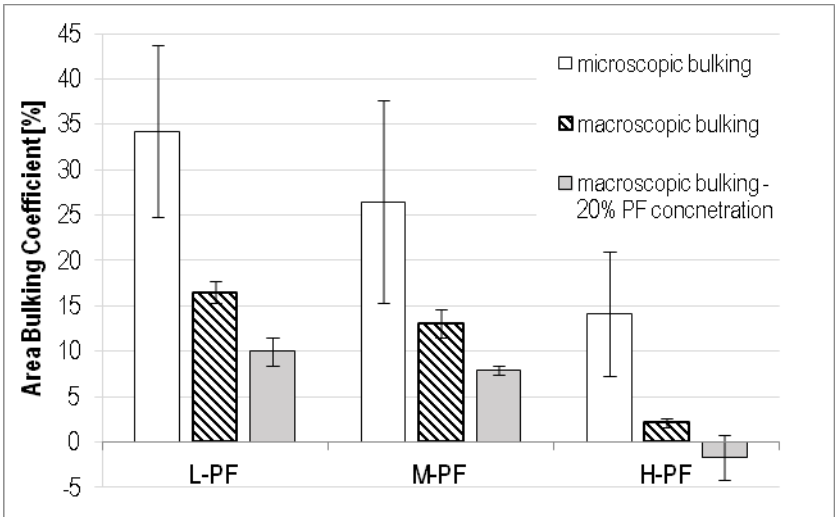

Fig. 1.
Area bulking coefficient (ABC) of the PF treated beech cell walls (microscopic bulking) and of PF treated beech blocks (macroscopic bulking) at a PF concentration of L-PF 45%, M-PF of 45% and H-PF 45% and wood blocks at 20% PF concentration (macroscopic bulking – 20% PF concentration).

Table 2

Weight percent gain (WPG) after curing and in the end of the third soaking-drying cycle, leaching coefficient (LC) and the ASE of beech blocks treated with the various investigated PF at various PF concentrations as well as the anti swelling efficiency (ASE) after each cycle*

PF type	PF concentration [%]	WPG after curing [%]	WPG after the 3rd cycle [%]	LC [%]	ASE 1st cycle [%]	ASE 2nd cycle [%]	ASE 3rd cycle [%]
L-PF	45	53.5 (3.0)	51.1 (3.0)	4.6	77.4 (2.2)	72.3 (1.2)	70.6 (1.2)
	20	26.3 (1.0)	24.3 (1.0)	8.4	65.1 (1.1)	62.8 (2.3)	58.6 (3.1)
M-PF	55	59.3 (2.2)	56.0 (6.9)	5.8	69.4 (3.1)	65.7 (1.3)	63.7 (3.5)
	20	25.1 (0.5)	24.1 (6.9)	4.2	60.5 (1.1)	55.6 (1.3)	53.1 (1.3)
H-PF	45	46.9 (4.3)	33.6 (0.5)	39.6	35.5 (2.2)	-2.1 (2.7)	-11.6 (2.9)
	20	22.2 (3.7)	17.4 (3.6)	27.2	23.6 (2.6)	-0.3 (3.1)	-6.3 (4.5)

*standard deviation in paranthesis

The dimensional stability for macroscopic wood blocks and microscopic cell walls after the first water soaking is expressed by the ASE. Results are displayed in Fig. 2 showing that the macroscopic ASE of the wood blocks treated with higher concentrations of L-PF (77%) and M-PF (69%) display a similar ASE range like the ASE on microscopic level (L-PF 86% and M-PF 74%). Furthermore, L-PF displays higher ASE values at microscopic and macroscopic level compared to the ASE of M-PF treated wood. Similar findings were described by Deka and Saikia (2000) and Anwar et al. (2009) claiming similar macroscopic ASE values for various wood species treated with commercial low molecular weight PF. However, significantly lower ASE were obtained from H-PF treated samples. Macroscopic ASE were determined with 35%, whereas the microscopic ASE was about 70%. As a result, H-PF treated beech wood displays a considerably higher microscopical dimensional stability compared to the macroscopic dimensional stability. However, a high variation was obtained for the microscopic ASE values and, thus, also for the SC at microscopic scale. Similar trends were mentioned by Buchelt et al. (2012) for furfuryl alcohol treated beech cell walls. These authors explained the finding with the varying restriction of swelling caused by surrounding cells and the varying chemical composition of each cell wall. Additionally, the degree of penetration of the PF into a single cell wall remains unknown and might also contribute to the high variation of the measured cell walls. Thus, the obtained microscopical ASE values should be interpreted with caution. Nevertheless, a trend is observable showing that H-PF can basically increase the dimensional stability due to cell wall penetration. Thus, dimensional stability might be mainly evoked by the PF molecules which are small enough to penetrate into the cell walls (Furuno et al. 2004, Anwar et al. 2009) and minor due to PF residuals in the cell lumina which are suspected to limit liquid flow into the wood substance. However, during the exposure of the treated wood to cyclic soaking and drying stress it could be shown that the macroscopic ASE further decreases for all applied PF types at all concentrations (Tab. 2).

As discussed for the leaching of the various PF, where the H-PF display a high LC during the leaching cycles, the macroscopic ASE of H-PF treated wood resulting in negative values after the second leaching cycle. At the same time, the L-PF and the M-PF show only a slight decrease of their initial ASE (appr. 7% for both applied PF concentrations). This supports additionally the hypothesis of an insufficient penetration of the H-PF into the cell wall. Additionally, after the first soaking, residual PF

in the cell lumina hinder the liquid flow of the water through the wood substance. This effect becomes poorer through leaching of the PF. This indicates that not only PF inside the cell walls affects the dimensional stability, but also PF inside the lumina could initially limit liquid flow and affects dimensional stability. However, these effect is not persistent. Furthermore, more work should be carried out on leaching of the certain PF out of single cell wall and its effect on dimensional stability.

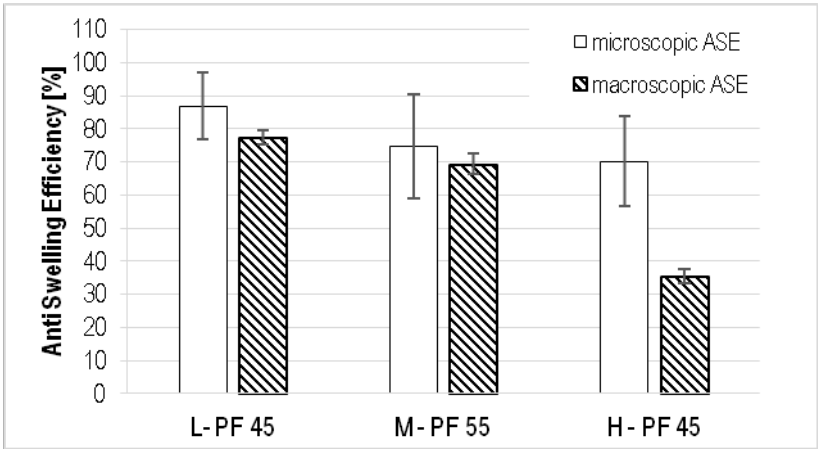

Fig. 2.
Anti swelling efficiency of the PF treated beech cell walls (microscopic ASE) and of PF treated beech blocks (macroscopic ASE). Where the number stands for the PF concentration of the certain PF.

CONCLUSIONS

In this study, three PF with varying molecular weight were investigated in respect to their potential to improve dimensional stability of beech wood. According to the obtained results it can be concluded that:

- L-PF and M-PF can penetrate to high degree into the wood cell wall, whereas H-PF basically have the potential to penetrate wood cell walls, but it seems that flow into the wood substance at macroscopic level is limited.
- Dimensional stability of beech treated with L-PF and M-PF is improved in the same extend at macroscopic level and on microscopic cell wall level, whereas H-PF displays clearly poorer dimensional stability on macroscopic level then on microscopic level. It can be concluded, that penetration and thus, sufficient cross linking of the smaller molecular weight fractions in the cell walls are the decisive factor for dimensional stability.
- During three soaking-drying cycles, only low amounts of L-PF and M-PF are washed out and corresponding, at the same time, decrease of the macroscopic dimensional stability is also affected to a small extend.
- Contrary to the L-PF and M-PF, H-PF is leached out during the three soaking-drying cycles in high amounts and simultaneously the dimensional stability results in negative values. This indicates insufficient interaction with the wood cell wall components. Furthermore, that PF residuals in cell lumina might initially increase dimensional stability, but this effect might not be persistent.

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INFLUENCE OF COATING FORMULATION ON PHYSICAL-MECHANICAL PROPERTIES

Daniela TESAŘOVÁ
Mendel University in Brno, Faculty of Forestry and Wood Technology
Zemědělská 1, 61300 Brno, Czech republic
Tel: 00420545134170, e-mail: tesar@mendelu.cz

Petr ČECH
Mendel University in Brno, Faculty of Forestry and Wood Technology
Zemědělská 1, 61300 Brno, Czech republic
Tel: 00420545134177, e-mail: cech.007@seznam.cz

Josef HLAVATÝ
Mendel University in Brno, Faculty of Forestry and Wood Technology
Zemědělská 1, 61300 Brno, Czech republic
Tel: 00420545134312, e-mail: hlavaty@seznam.cz

Abstract

The aim of this contribution is the investigation of the influence of coating formulation on physical – mechanical properties; especially on the hardness of finished surfaces, the adhesion of the lacquers films to wooden substrates, the resistance of finished surfaces to impact, the resistance of finished surfaces to mechanical damage - the impact tests. Tensile tests on free coating films of the same lacquers have also been measured. In this study we investigated transparent nitrocellulose, polyurethane and UV curing high solid acrylic lacquers. The results reached in this study have confirmed the relationship between elastic modulus and the stress at break of free lacquer films and the physical-mechanical properties of finished surfaces.

Key words*: physical—mechanical properties; maximum force at break; tensile stress; hardness.*

INTRODUCTION

Wood, as a natural construction material in use, needs the protection of by coating films to keep its beauty, colour and integrity. Wood movements due to swelling and shrinking induce stress to the surface coating. Results of this stress, between the wood surfaces and coating films, are the decreasing quality of physical–mechanical properties. The aim of the study is based on the hypothesis that this is caused by the relationship between the physical-mechanical properties and the tensile properties of lacquer films.

In this contribution we present there are presented the results of our investigation of the influence of coating formulations on the physical-mechanical properties of furniture finished surfaces. Improving the durability and improving of the physical-mechanical properties of finished furniture surfaces is essential for prolonging the life of the furniture. The conclusions of this study have great influence, not only on the coating performance but also on the quality of wooden-based products. They can help to improve the physical-mechanical properties of finished surfaces of wood because it is supposed that he tensile stress of coating films has significant influence on the performance of finished surfaces. The tensile properties of coating films have not been assessed yet in correlation with physical-mechanical properties.

OBJECTIVE

The aim of this study was to identify the relationship between influence of tensile strength during the tensile stress at break of the tested lacquer coating films, and the quality of mechanical-physical properties of the finished surfaces coated with the tested lacquers.

MATERIAL, METHODS, EQUIPMENT

Five different lacquers were tested for the evaluation of the influence of different test parameters; this means that five different resins have been investigated.

1. nitrocellulose lacquer,
2. top solvent polyurethane lacquer
3. basic solvent polyurethane lacquer

4. acrylic water borne lacquer
5. UV curing high solids acrylic lacquer

Preparing the samples:

Each one of the tested coating materials was applied to a sample of chipboard veneered with pine veneer. The amount of coating lacquer varied from 40g/m^2 (UV curing high solids acrylic lacquer) to 300 g/m^2 (solvent polyurethane, basic solvent polyurethane lacquer, water borne lacquer and nitrocellulose lacquer).

Each one of the tested coatings was applied to on polyterephtalate foil by using the laboratory film applicator. The coatings were removed from the foil, in controlled environmental conditions immediately after drying/curing took place. The tested films were carefully detached by hand and cut size (using a scalpel). The size of the films was 10 mm x 50 mm. The specimens were oriented longitudinally.

Test methods and standards used

- Adhesion Paints and varnishes cross-cut standard ČSN EN ISO 2409
- ČSN 910277 Furniture. Testing the furniture surface coating. Method of determining the surface impact resistance
- BS 3962 part 6 The resistance of finished surfaces to mechanical damage - the impact tests
- ČSN EN ISO 2815 Buchholz indentation tests
- Tensile tests were performed using a test device by of the company Instron 3365 Machine Serial Number Locator with measurement software Blue hills
- ČSN EN ISO 527-3 Determination of tensile properties Part 3 The conditions for films and foils
- ČSN EN ISO 527-1 Determination of tensile properties Part 1 General principles

RESULTS AND DISCUSSION

On the figure numbers 1, 2, 3, 4, 5 and 9 we can observe the physical-mechanical properties of the tested finished surfaces of the chipboard samples veneered with pine veneer. In figure numbers 6, 7, 8, 10, 11 and 12 are the results of measuring the tensile stress at break, force at break and elongation of tested lacquer films. The mean values and standard deviations of assessed properties were determinate and calculated for elongation of the sample of the lacquer film in maximum force (F_{max}). The charts have shown the behaviour of the coating films during the tensile tests. Great differences in behaviour during testing have appeared especially among the water borne lacquer films and UV coating films. The stress curves of tested each one tested coating materials were very different in dependence of used resin.

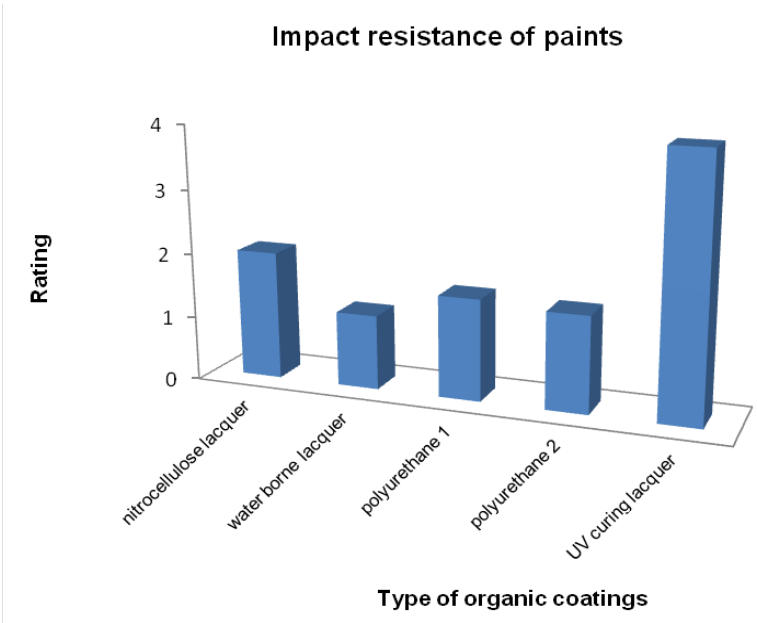

Fig. 1.
Impact resistance of paints.

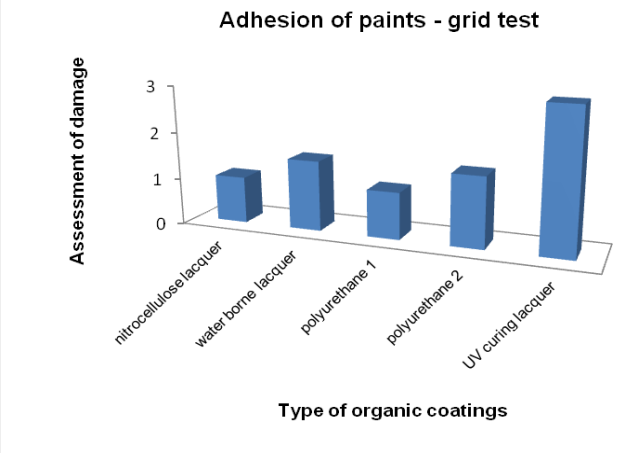

Fig. 2.
Adhesion of paints – grid test.

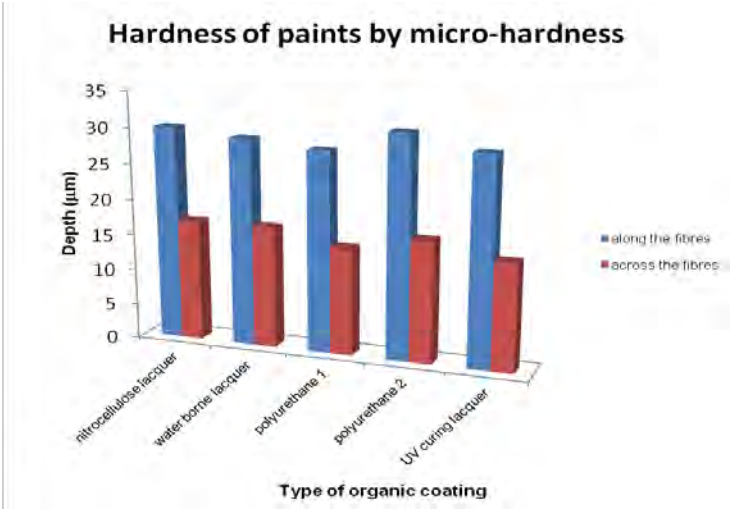

Fig 3.
Hardness of paints – micro-hardness

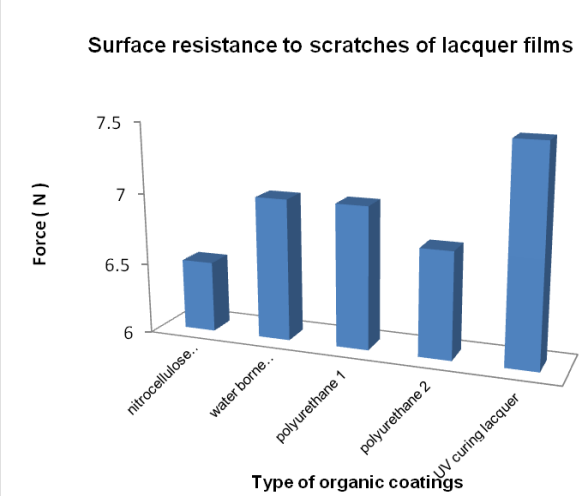

Fig. 4.
Surface resistance to scratches of paints.

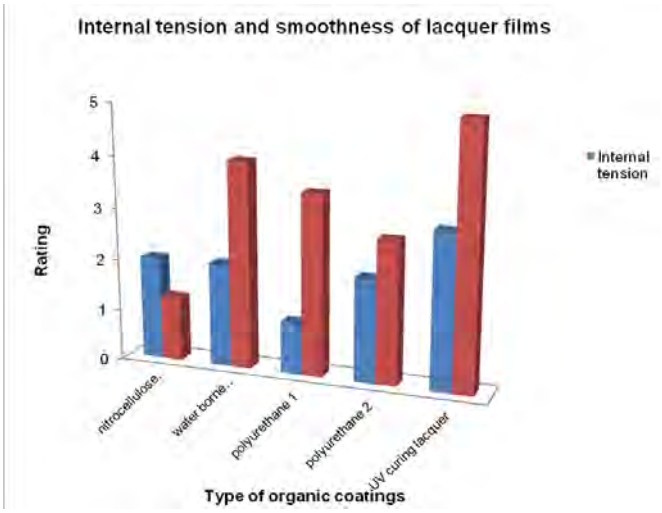

Fig. 5.
Internal tension and smoothness of lacquer films.

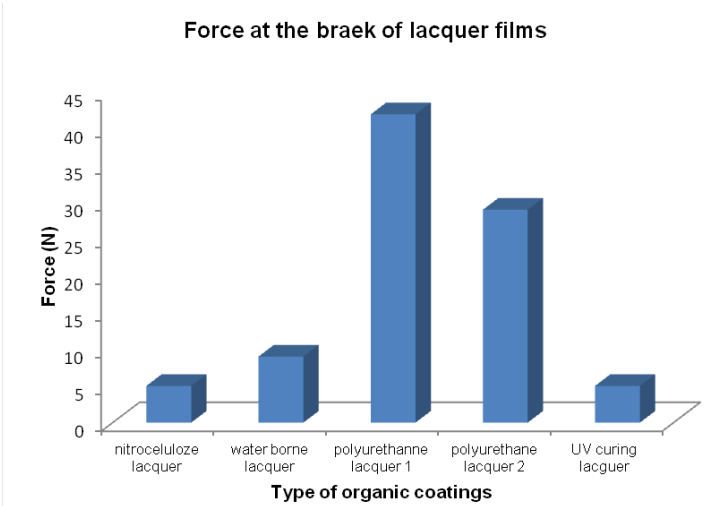

Fig. 6.
Force at the braek of lacquer films.

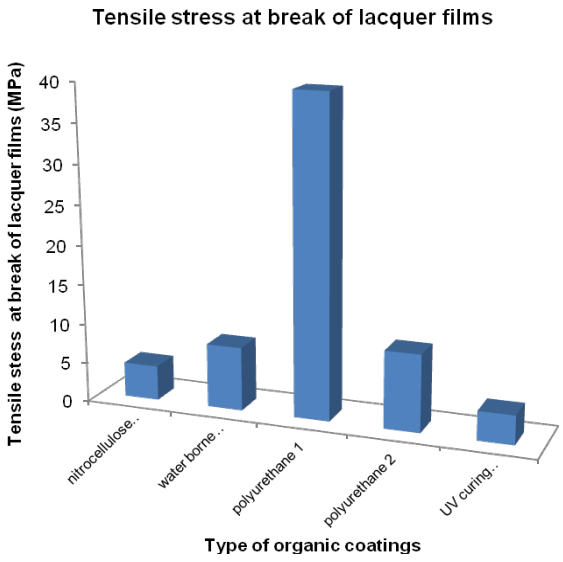

Fig. 6.
Tensile stress at the break of lacquer films.

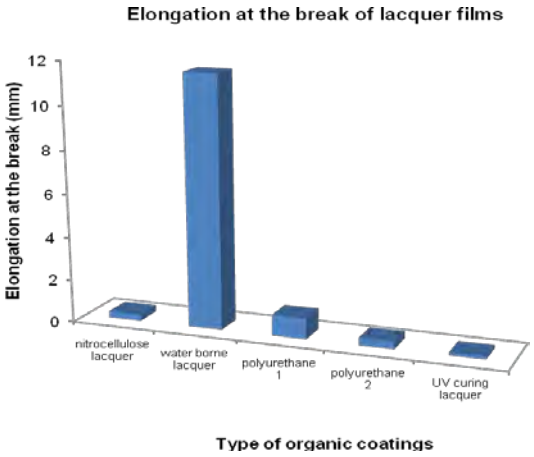

Fig. 7.
The elongation at the break of lacquer films.

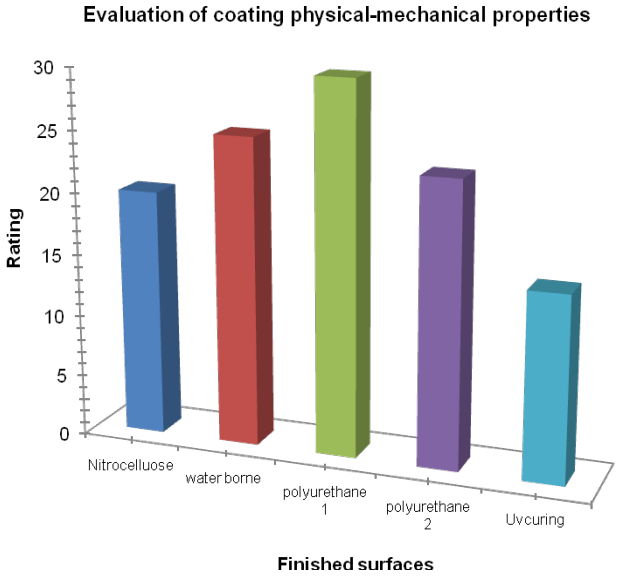

Fig. 8.
Evaluation of finished surfaces physical-mechanical properties.

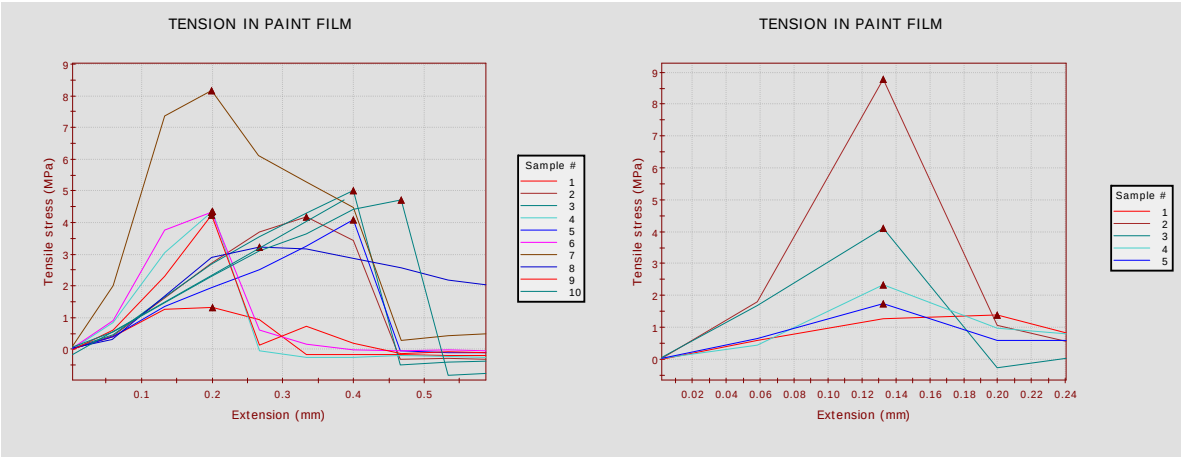

Fig. 9.

a-Tensile tension in samples of lacquer films; nitrocellulose.

b –Tensile tension in samples of lacquer films UV curing lacquers.

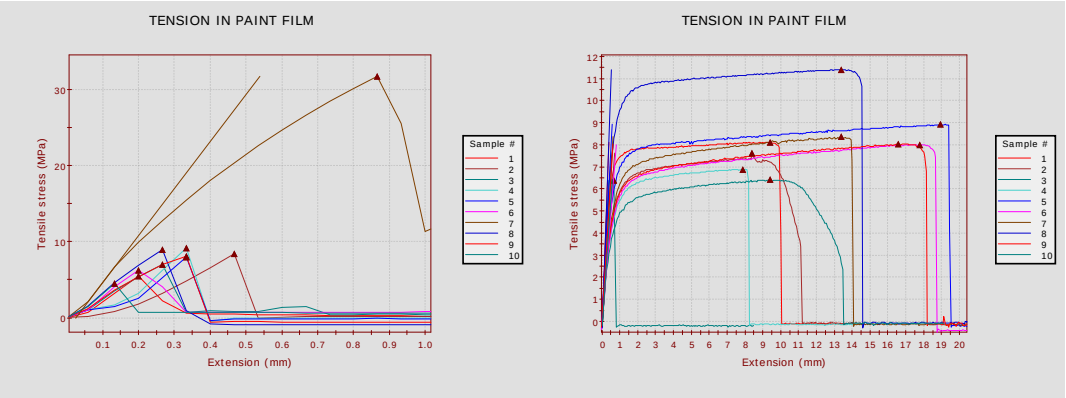

Fig. 10.
a –Tensile tension in samples of lacquer films polyurethane-2K. ***b –Tensile tension in samples of lacquer films water borne lacquer.***

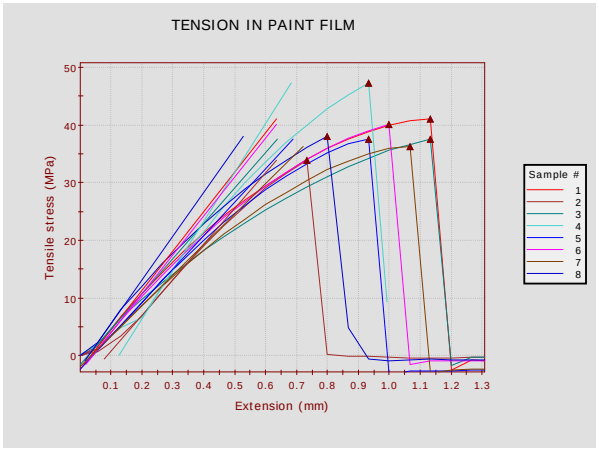

Fig. 11.
Tensile tension in samples of lacquer films of polyurethane-1K.

CONCLUSIONS

In this contribution we achieved very important results. When we have compared the results of physical –mechanical properties of finished surfaces and the results of tensile stress at break of lacquer films, we found the relationship between tensile stress stresss and physical-mechanical properties. The results of physical-mechanical properties are summarized in figure 9, the results of the assessment of tensile stress of coating films are expressed in figure 6 and the forces at break of lacquer films are shown in figure 7, and when we put them together we compared all results. These achieved and compared results confirmed our hypothesis about the relationship between the physical-mechanical properties of lacquers films and the ultimate tensile stress of free coating films.

The coating film of the polyurethane lacquer 1K delivered the best results during the investigation of physical-mechanical properties of finished surfaces and kept the highest tensile stress at the break of lacquer films. UV curing lacquer films provided the worst results in both of the testing methods (physical –mechanical). When we compared both of figures 6 and 7 to the results in figure 9, we could see the relationship between the tested physical-mechanical properties of finished surfaces and lacquers films made from the same coating materials.

The harmonization of the tensile test conditions for free wood coatings is mandatory before talking about threshold values or limits for mechanical properties. This study has shown that it is very important to investigate the tensile stress of free coating films during the development of coating materials.

ACKNOWLEDGEMENT

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WATER PERMEABILITY OF TWO DIFFERENT WOOD LASURES

Jozef VALENT
eng. - University of West Hungary, Faculty of Wood Sciences, Institute of Product Design and Manufacturing , Sopron, Hungary
Address: Bajcsy-Zs. 4., 9400 Sopron, Hungary
E-mail: valent.jozef@citromail.hu

Eva PAPP
eng. - University of West Hungary, Faculty of Wood Sciences, Institute of Product Design and Manufacturing , Sopron, Hungary
Address: Bajcsy-Zs. 4., 9400 Sopron, Hungary
E-mail: papp.eva@fmk.nyme.hu

János PALLA
University of West Hungary, Faculty of Wood Sciences, Sopron, Hungary
Address: Bajcsy-Zs. 4., 9400 Sopron, Hungary
E-mail: tai8@freemail.hu

Csilla CSIHA
dr. eng. - University of West Hungary, Faculty of Wood Sciences, Institute of Product Design and Manufacturing , Sopron, Hungary
Address: Bajcsy-Zs. 4., 9400 Sopron, Hungary
E-mail: cscsiha@fmk.nyme.hu

Abstract

Due to recent developments more and more new coatings appear on the market being proposed by the manufacturer for outdoor use. One major group of new products targets the reduction of harmful emissions (RDS) and thus instead organic solvents water is used as dispersing agent and also new resins in their composition. In the present study a classical alkyd based solvent-borne thin layer lasur was compared with a water-borne thixotropic wax, by monitoring water uptake during long term soaking. The samples treated with wax showed 4% mass increase relative to the initial mass after 156 hours, whilst the solvent-borne lasur treated samples showed 4% mass increase after 456 hours. Based upon the upper considerations the solvent-borne lasur performed 3 times better than the wax based one. It is supposed that the water permeability behaviour can be connected to lifetime performance also. Molecule size investigations are needed in order to explain the results.

Key words: *solvent-borne; water borne lasures; tixotrophic wax; Birch; soaking; water permeability.*

INTRODUCTION

Water-borne paint systems and as such water-borne lasures also, were developed in a legislative environment strongly driving paints towards VOC free or at least reduced VOC varnishes. In this term the water-borne systems are much younger than the solvent-borne ones. Their overall performance has improved since they were first introduced in the early 1970s, but the products still require special handling (Cox 2003). Unfortunately water brings operational disadvantages and in the case of wood this includes increased grain raising, and problems arising from the high latent heat of water particularly under industrial conditions (Bulian – Graystone 2009). Alcohol does not raise the grain of the wood as much as waterborne stains (Cox 2003). On the other hand the use of water-borne systems can considerably reduce the emission of organic compounds (VOC) during surface treatment, drying and also from the product (Prieto-Kiene 2007). A further step towards environment friendly coating systems can be the use of waxes.

Wax additive is an important functional additive for waterborne wood coatings. There are different kinds of wax additives available for wood coating films. Shengwen and colleagues reported that after being added in the wood coatings, the wax particles were homodispersed in the coating films, and could improve the tackiness resistance and the scratch resistance of coating films. But in the same time large size of wax particles, affected negatively the gloss of the film (Shengwen 2007). Generally waxes are supposed to be hydrophobic and thus acquiring the same properties as well to the objects treated with. Lesar et al. (2011) also presumed, that wax treatment will reduce water

uptake and thus reduce or slow down photo-degradation processes of Norway spruce specimens. Three differing types of waxes were used: an emulsion of montan wax (LGE), an emulsion of polyethylene (WE1) and an emulsion of oxidized polyethylene (WE6) wax. Besides FTIR spectroscopy and SEM analysis the water uptake after artificial ageing was also evaluated. They concluded that: impregnation of wood with waxes influences the performance of wood during artificial accelerated weathering. Treatment of wood with high loadings of wax reduces moisture absorption by wood subjected to accelerated weathering and restricts photodegradation of wood. Among selected waxes, montan wax was the most effective at restricting photodegradation. However, wax treatments reduce the photodegradation only to certain extent. Scholtz et al. (2010) did investigations on migration and deposition of hot melting wax in wax-treated pine sapwood (*Pinus sylvestris* L.) and beech (*Fagus sylvatica* L.). Three waxes were used, which did not show distinct differences in their deposition patterns. An intensive wax deposition could be observed within the vessels, tracheids, and fibers. In *P. sylvestris* the ray tracheids were penetrated in a lateral wood penetration process, from the outer to the inner wood. In general, no wax penetration was visible within the parenchyma tissue and epithelium cells. Cracks were detected within the wax deposits as well as secondary microcapillaries, which were visible between the deposits and the cell walls. Esteves et al. (2012) did preliminary investigations on *Pinus pinaster* wood impregnated with paraffin to different levels using a hot-cold process. Weight gain, density, equilibrium moisture and dimensional stability (ASE) at 35% and 65% relative humidity and termite durability against *Reticulitermes grassei* (Clément) were determined. The best anti shrinking efficiency (ASE) was obtained for a combined treatment at 180ºC (4h) and 61% WG. The preliminary tests with paraffin impregnation showed that wood has lower equilibrium moisture, higher dimensional stability.

In Hungary, those wood coating materials which are proposed for outdoor use are called lasures, mostly deriving from the German terminology. Lasures due to their specially designed molecule size which allows water vapour to penetrate the layer are the most appropriate varnishing materials for outdoor exposure (Posch 1996). They show further special properties as they protect the wood surface from liquid water, but in the same time allow the water vapour diffusion through the coat and also protect the wood surface from the UV radiation, ensureing this way a longer service life for the coat than the one of the traditional oil based glazes. Lasures are produced with differing amount of resin and they are distinguished accordingly as: thin layer and thick layer lasures. According to the layer they are used in and also to the type of protection they exhibit, they are called sometimes intermediate isolating,- protective,- fiber binding etc. lasur layers. For the recent study an acrylic acid ethyl ester type wax based lasur was used, described as system deeply penetrating the wood, but after several application steps, in last layers forming also a film. From the range of available lasur types the thin layer one was chosen for comparison, as they behave in the same way as the wax described above: they penetrate the wood tissue and after several steps of application they also form a comprehensive layer. The principle of comparison was that after several steps of lasur application the dry amount to be the same. This idea was also supported by the description given by the manufacturers, as they gave suggestion on several steps of application without strictly limiting the upper border. The all over study on comparing the water uptake of wood samples coated with water-borne wax and solvent borne conventional lasur was conducted in order to describe the possible differences in the behaviour of the two differing coating systems.

MATERIAL AND METHOD

The wood species chosen for sample preparation was Birch (*Betula pendula).* When choosing Birch wood for sample preparation, two considerations were made: it is a diffuse-porous wood species, with relatively homogeneous anatomic structure, having earlywood and latewood vessels of closely constant diameter, and deriving from the anatomic structure even water uptake could be supposed. The average density of Birch samples was 688 kg/m^3 (st dev. 11kg/m^3), after brought from the factory, all were conditioned at 20^{o}C±2^{o}C and 60±% relative humidity in laboratory, having in average 9,2 MC.

Sample preparation:

Boards of 800x100x20mm all kiln dried, of selected furniture quality, with selected homogeneous, tangential and partly radial cut Birch boards were used for the present study. Prior to be cut to the testing size, the whole boards (2/lasur type) were coated using a roller. Two types of lasur were used: an acrylic acid ethyl ester type wax based lasur (Pigrol), and a thin layer solvent-borne alkyd based lasur (Xyladecor). The wax was described as system deeply penetrating the wood,

but after several application steps, in last layers forming also a film. From the range of available lasur types the thin layer solvent-borne one was chosen for comparison, as they behave in the same way as the wax described above: they penetrate the wood tissue and after several steps of application they also form a comprehensive layer. After each step of lasur application the surfaces were dried according to the manufacturer's recommendations. The drying time of wax was approx. 2 hours, whilst the drying time of solvent-borne lasur was approx. 24 hours. Both finishes were applied in several steps, after each step the surfaces were dried, the dry samples were weighted, and compared with the untreated status the applied dry amount of finish was calculated. In case of wax 146g/m2,- whilst in case of solvent-borne lasur 143g/m2 dry finish was applied.

As originally from the factory 800mm long samples were brought, they were cut to size of 150x70x20mm according to EN 927-5 (2006). The five untreated surfaces of the samples were sealed using transparent silicon, overlapping even the test face by 2mm. Untreated control samples were also prepared in the same size. According to the standard the treated samples were supposed to a pre-soaking in distilled water, as according to previous observations the water permeability of some types of coatings can change markedly during a relatively short period of exposure to water. For such coatings the values of water permeability after a short period of contact with water may not be representative of those obtained during long term service. Thus a repeated pre-soaking is suggested, aiming leaching of some leachable substances: 24 hours soaking of the test face, 3 hours drying at room temperature, 3 hours drying at 50^{o}C and 18 hours at room temperature (2x).

Treatment:

All three types of samples were weighted to the nearest 0,01 g, mass recorded, and were laid with the unsealed face down on a special tray, containing distilled water, not covering the samples, but ensuring a continuous immersion of the test face. The level of the distilled water was kept nearly the same by manually replacing the consumed amount.

Testing:

Water uptake showing the water permeability of the lasures was monitored by mass measurements. Contrary to the suggestion (72 hour) of EN 927-5 mass measurements were continuously performed at 0; 1; 2; 3; 4; 5; 6; 7; 8; 9; 11; 13; 15; 17; 19; 21; 23; 24; 27; 39; 42; 46; 50; 62; 67; 72; 84; 90; 96; 108; 120; 132; 144; 156; 168; 180; 204; 216; 228; 240; 252; 264; 276; 288; 300; 312; 324; 336; 348; 360; 372; 384; 396; 420; 432; 444; 456; 468; 480 hours.

RESULTS AND DISCUSSION:

Evaluating the water uptake graphs a surprising statement can be done. Contrary to the expectations the samples treated with water-borne wax showed higher water uptake from the beginning than samples treated with solvent-borne lasur. In both cases the water uptake was much lower than the water uptake of untreated samples, showing that the wax also acted against taking up water, but its measure of resistance was lower than the one of the solvent-borne lasur.

The samples treated with wax showed 4% mass increase relative to the initial mass after 156 hours, whilst the solvent-borne lasur treated samples showed 4% mass increase after 456 hours. Based upon the upper considerations the solvent-borne lasur performed 3 times better than the wax based one. It is supposed that the water permeability behaviour can be connected to lifetime performance also, in terms that a more intense water uptake is supposed to cause the deterioration of the coat film in shorter time. It was stated that no sample reached the saturation point during the 456 hours of soaking, although the increase was very slow: for untreated samples in 12 hours approximating for example 0,7521g (168 hour and 180 hour), for wax treated samples in the same time period 0,1221g, whilst for solvent –borne lasur treated samples 0,04g. Based on theoretical considerations the reason of the high water uptake in case of the wax was supposed to be due to a high molecule size. In a further study the molecular weight of the two lasures is going to be investigated.

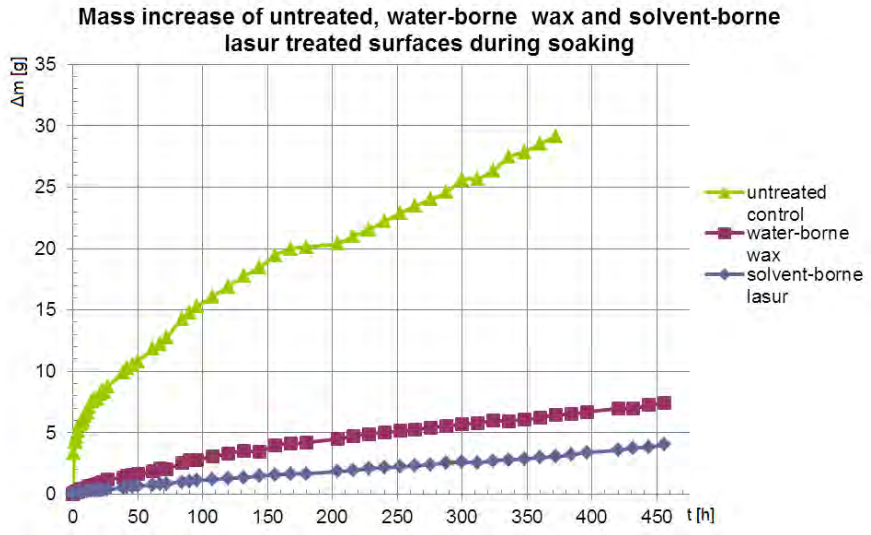

Fig. 1.
Mass increase of untreated, water-borne wax and solvent –borne lasur treated surfaces during soaking.

CONCLUSIONS:

Evaluating the water uptake graphs the following statement was done: contrary to the expectations the samples treated with water-borne wax showed higher water uptake from the beginning than samples treated with solvent-borne lasur. The samples treated with wax showed 4% mass increase relative to the initial mass after 156 hours, whilst the solvent-borne lasur treated samples showed 4% mass increase after 456 hours. Based upon the upper considerations the solvent-borne lasur performed 3 times better than the wax based one. It is supposed that the water permeability behaviour can be connected to lifetime performance also. Molecule size investigations are needed in order to explain the results.

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COMPARING DURABILITY OF WOODEN PRODUCTS AGAINST MICROCEROTERMES DIVERSUS IN REGIONS OF JIROFT AND SISTAN OF IRAN

Ali BAYATKASHKOLII
University of Zabol, College of Natural Resources, Department of Paper Wood Technology and Sciences, Zabol, Iran
Tel: 00989177153975, Fax: 00985422232600, E-mail: ali.bayatkashkoli@gmail.com, ali.bayatkashkoli@uoz.ac.ir

Batoul KAMESHKI
University of Zabol, College of Natural Resources, Department of Paper Wood Technology and Sciences, Zabol, Iran
E-mail: batoolkameshki@gmail.com

Soltan RAVAN
University of Zabol, College of agricultural, Department of Plant Protection, Zabol, Iran
E-mail: rbayatkashkoli@yahoo.com

Mohammad SHAMSIAN
University of Zabol, College of Natural Resources, Department of Paper Wood Technology and Sciences, Zabol, Iran
E-mail: mohammadshamsian1@hotmail.com

Abstract

Wood-based panels are used at inside and outside of various building, and termites feed mostly from these lignocelluloses materials. The aim of present study identifies biological resistance of four wooden products that include medium density fiberboard, 3-plywood and particleboard against Microcerotermes diversus termite via field method. Degradation grading the termite attack was performed in accordance with the ASTM D 1758-06 specifications. This research was conducted in Sistan region and Jiroft for 15 months. On the base of this result, all testing samples have been degraded. Both weight loss and grading values indicated that the 3-plywood and medium density fiberboard had the lowest and highest degradation, respectively. The weight loss and grading values of wood based panel specimens against the Microcerotermes diversus attack is the same. Field observations showed that the degradation of composite specimens located at Sistan are similar to Jiroft, except of particleboard specimen degradation.

Key words: *biological resistance; wooden products; Microcerotermes diversus termites.*

INTRODUCTION

The wooden products and cellulosic industries have had considerable growth and development. The increasing consumption of wooden products that is followed by population growth, and demand extension has increased consumption of raw wood. Concerning ascending trend of consumption of wood and its products and limited wooden sources, the lack of raw wood is very evident. Wooden composites have been used extensively instead of raw woods in structural. A large amount of wood products that are in contact with soils degrade and many losses are incurred to the economy. Identification of resistant wood products against termites can be a good guide for selection of wood products in different consumptions. Studies conducted on durability of productions against termites and in field condition are limited, but studying the durability of wood and wood products in vitro has a relatively good and generally they associate with durability of woods against fungi.

Cellulose being the principal food of termites, wood and wood products such as paper, fabrics and wood structures are avidly consumed (Peralta et al. 2004). A five year test in Japan showed that softwood 3-plywood, particleboard and oriented strand board were not resistant against termites attack (Tsunoda 2008). Studies indicated that the first fracture on the glue line resulted in start of degradation of wood-based composites against biological degradations (Carll and Highley 1999; Kartal and Clausen 2001; Vick et al. 1996; Wagner et al. 1996).

The ability of white and brown rot fungi and termites to decompose MDF consisting of different wood species by measuring weight loss evaluated. MDF and wood specimens were also bioassayed

against the eastern subterranean termite, Reticulitermes flavipes (Kollar) in order to determine termite resistance of the specimens. MDF specimens made from beech and mixed furnish showed decreased weight losses from termite attack after 4 weeks. However, none of the MDF specimens were resistant to termite attack. In severe conditions, the MDFs may require the incorporation of chemical biocides prior to board production for increasing the resistance of MDF to termite attack (Kartal and Green 2003). Laboratory termite resistance tests showed that all samples containing boron compounds had greater resistance against termite attack compared to untreated MDF samples. As chemical loadings increased, termite mortalities increased, and at the same time the weight losses of the samples decreased (Usta et al. 2009).

Particleboard and medium density fiberboard (MDF) panels were produced using stone pine (Pinus pinea) cones, which were mixed with either wood particles or fibers from pine and beech wood at various ratios. Specimens were also subjected to subterranean termites, Coptotermes formosanus, according to the JIS K 1571 standard test method for 3 weeks. No increased resistance was observed in the specimens exposed to the termites. In some cases, the specimens containing pinecone furnish had greater mass losses compared to the control specimens (Kose et al. 2011). Five kinds of commercially available wood-based composites (softwood plywood, hardwood plywood, medium density fiberboard, oriented strand board, and particle board) post-treated with alkaline copper quat and copper azole were exposed to decay and subterranean termite activity for three years. Both biological attacks developed with time. Untreated medium density fiberboard and particle board were highly resistant to field conditions during the 36 months. Untreated oriented strand board, hardwood plywood and softwood plywood were the least resistant composite types (Tascioglu et al. 2013).

The wood has a special structure and consists of carbon, hydrogen, and oxygen and it is considered as a natural organic material, it can degrade easily by different biological degradations. Termites in their geographical limits threat building timbers, goods made from wood, paper, and dress, for special types of arable trees and plants and some types of plastics. Termites have an extensive distribution throughout the world and they damage wooden products. All wooden composites have been made by less durable woods or their coats have small holes are sensitive to attack of termites especially when they are subject to soil. Wooden materials are degraded by termites even when are apparently dried. It is important to identify wooden products resistant against termite.

OBJECTIVE

The main objective of the present research was to identify the wood-based panels resistant against termite *Microcerotermes diversus*.

MATERIAL, METHOD, EQUIPMENT

Termites and geographical distribution

The geographical range of termites is mostly in north of Africa, Middle East and southwest of Asia such as south of Iran (Kerman and Sistan & Baluchistan) and south of India. Generally, most of termites are living in tropical and rainy jungles but their activity is higher in unstable jungles and plains under cultivation meaning where there are farms. The number of species and termites will reduce rapidly outside the tropical regions or where the earth height causes reduction of temperature. Many termites are living in warm soils in the world. Some of them are hidden under the soil surface and are invisible while others have obvious mound above the land surface. Some of such nests are only few centimeters high while others are 9m high.

Termite genus Microcerotermes diversus is living in countries such as Iran, Iraq, Kuwait, Oman, Arabia, and United Arab Emirates. This insect has been reported in provinces of Khuzestan, Boushehr, Sistan & Baluchistan, and Yazd in Iran (Habibpour 2008).

Termite genus Microcerotermes diversus

This family includes a ratio four fifth of all types of termites in the world. Concerning morphology, these are wood feeding termites and often they are living under ground or they make mounds (Epigeal nests) but a few of them will built above ground, and always connected to the ground (such as arboreal nests). Termites have very strong and abrasive parts in their mouths. They have antennas with 21-32 articulations. They have two membrane wings that are laid on their back during resting and the word isopteran is used for equality of their wings. These insects are known as isopteran and have social life and are called termites. Its head is yellowish brown, their antenna is light yellow, and their mandibles are reddish dark brown and at the top they are a bit black, the part under the front is darker than the head. Top mandibles are sharp and a bit shorter than head capsule and their shape is sickle with thin and sharp lobes. This genus is living in closed places where the air is mild and relative humidity is 50%. The light will not penetrate in mounds of termites and the amount of

carbonic gas is high and the amount of oxygen is low. The temperature of colony is between 18 and 22^0C (Harris 1971).

Geographical position of the regions

This research was conducted at two locations for 15 months in field condition. Ghaemabad village in Sistan region located at south east of Zabol, and Miandeh is located at the south of Jiroft. Jiroft is located at the south of Kerman province of Iran in a plain in 28 degrees, 40min in north and 57 degrees, 44min in east to Greenwich noon. Jiroft climatic has changed from warm status to cold and temperate. The penetration of humidity of Indian Ocean causes torrential rain. In summer, a very warm wind (known as Hoosha and Kouhbad) blows from mountains of north and north east to Jiroft plain. The wind that sometimes lasts for 7 days reduces humidity in Jiroft. Mean precipitation is 82.0 mm per year and its temperature is between 6.2 and 39.6^0C. Sistan is located at the north of Sistan & Baluchistan province of Iran in a uniform plain in 31 degrees, 20min in north and 61 degrees, 39min in east to Greenwich noon. Zabol has a hot and arid climate. Mean precipitation is 6.59mm per year and its temperature is between 5.9 and 49^0C (Iran meteorological organization).

Field test

Holes were dug with 30cm deep and 50cm diameter. Cement rings have been used inside the holes in order that the soils do not fall on the samples. Specimens were randomly placed in the bottom of the holes so that the thickness faced the ground; they were then fixed in position using metal wire. Three types of wooden products including medium density fiberboard, 3-plywood and particleboard were degraded by termite genus Microcerotermes diversus in field method. Degradation grading and evaluating against the termite attack was performed in accordance with the ASTM D1758-06 specifications. The samples were placed in holes. Each sample was hung from the bar with wires and after putting samples in the hole, thickness of samples was in contact with the soil. They were visually observed per three months and the final observation was done after 15 months. Once specimens were positioned, the openings of the holes were covered with galvanized sheet covers. Dry weight of the primary samples before being degraded by termites has been estimated based on wet content (ISO 13061-1). At the end of the 15-month exposure, specimens were brought out of the holes to be weighted again for weight loss calculation. Weight loss of samples degraded by termites was measured based on dry weight in the last stage.

Statistical Analysis

The descriptive statistic of mode was used to determine differences between wooden products on the basis of degradation grading. Cluster analysis was performed to find similarities and dissimilarities between treatments based on more than one property (Ada 2013). Hierarchical cluster analysis, including dendrogram and using Ward methods with squared Euclidean distance intervals, was carried out by SPSS/18 (2010). One way variance analysis was used to determine significant difference between weight losses of wooden products at the 95% level of confidence. Grouping was then made between treatments by using the least significant difference (LSD) at 95% confidence level. Fitted-line plot between weight losses versus degradation grading were performed.

RESULTS AND DISCUSSION

Climate conditions and soil properties of field sites such as Electricity conductance (EC) are shown in Table 1.

Wooden products specimens were exposed to Microcerotermes diversus termite at two regions. The first visual observation was done after three months. The evaluation was done during 5 three month periods. After visual observations, the score of degradation was recorded based on ASTM standard. Results of statistical analysis are shown in Tables 2 as descriptive statistics (mode). The raw materials and adhesive of wooden products are presented at Table 2.

Results (mode) obtained from wooden products during 15 months in field condition have shown that the termites degradation is the difference at two regions, except of particleboard specimen degradation. The 3-plywood had the highest resistance against Microcerotermes diversus (Table 2). The final observation of samples in order of degradation amount is 3-plywood, particleboard and medium density fiberboard.

Table 1

Climate conditions at periods the study and soil properties are relate to Jiroft and Sistan regions.

Field site	Climate parameter	Periods the study				
		May June July	August September October	November December January	February March April	May June July
Jiroft	Average temperature	35.5	24	12	28	34
	Relative humidity	32	36	52	47	37
Sistan	Average temperature	32	30	11	39	33
	Relative humidity	27	28	49	38	27
	Soil properties					
	Type soil	pH	(EC) (ds/m)		Density (g/m3)	
Jiroft	Sandy, clay, loam	7.81	3.24		1.43	
Sistan	Sandy, loam	8.71	22.7		1.53	

Table 2

Degradation grading of the medium density fiberboard, 3-plywood and particleboard specimens in three-month intervals exposed to Microcerotermes diversus termites at regions of Jiroft and Sistan, based on the mode descriptive statistic (results of a 15-month field test), and also properties of wooden products.

Wood-based panels				Regions	Symbol name	Grade No. Degradation of three-month periods (Mode)				
Type	Density (g/cm2)	Raw materials	Adhesive							
MDF	0.77	hardwood	Urea formaldehyde	Jiroft	11	10	8	8	7	6
				Sistan	21	10	9	9	8	7
3-plywood	0.60	Poplar	Phenol formaldehyde	Jiroft	12	10	10	10	10	10
				Sistan	22	10	9	9	9	9
particleboard	0.78	Hardwood	Urea formaldehyde	Jiroft	13	10	10	8	8	7
				Sistan	23	10	9	8	8	7

Cluster analysis includes dendrogram based on the degradation grading values is shown in Fig. 1. Cluster analysis based on the degradation grading values showed that particleboard exposed to *Microcerotermes diversus* termite at Jiroft region (symbol name: 11) were clustered significantly different from the others wood-based panels. But, the weak impact of placement on the biological resistance of wooden products against Microcerotermes diversus termites is illustrated (Fig. 1). Also, the 3-plywood specimens against *Microcerotermes diversus* termites (symbol name: 12 and 22) were clustered significantly different from the others wood-based panels.

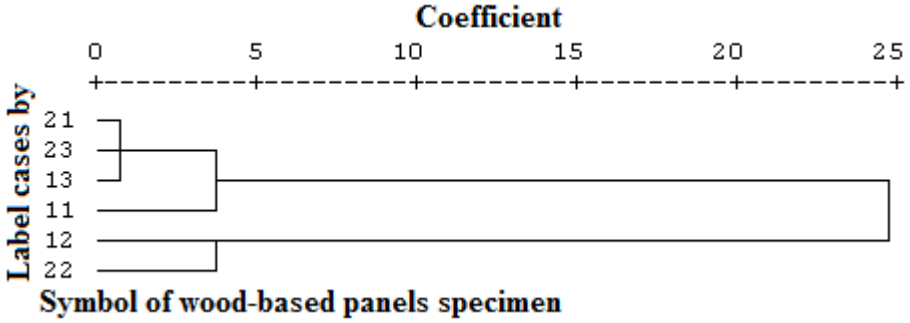

Fig. 1.

Cluster analysis of the wood-based panels based on the degradation grading values. Wooden products specimen symbol is present at Table 2.

In the first three months (July, August, September), the gradation only started on medium density fiberboard. Then, at the end of autumn and in the third three months (January, February, March), degradation stopped due to temperature drop at Jiroft. At the end of the third three months and start of the fourth three months (April, May, June) that is the season of mating for termites, degradation started again. On the other hand, the highest percent of degradation started from September, October, and November and stopped due to temperature reduction in December, January, and February meaning at the end of autumn and the beginning of winter and it again started at the middle of March at Sistan. In September, the samples were exited from holes for final observation after 15 months and degradation score was given to species during visual observations (Table 2).

Statistical analyses of the least significance difference shows that weight loss of wood-based panels have a significant difference between treatments at the 95% level of confidence. Percent of weight loss of composites and also, result of LSD multiple comparisons are seen in Table 3.

Table 3

The mean and standard deviation of weight loss and also, result of LSD multiple comparisons.

	one-way descriptive		multiple comparisons LSD			
Symbol name of Treatment	**Mean of weight loss**	**Standard Deviation**	**(I) Treatment**	**(J) Treatment**	**(I-J) Mean Difference**	**Sig.**
11	25.128	3.725	11	12	18.032*	0.000
12	7.097	2.830		13	15.691*	0.000
13	9.438	2.983		22	18.609*	0.000
			12	21	-14.177*	0.000
21	21.273	4.396		23	-13.615*	0.000
22	6.519	3.461	13	21	-11.836*	0.001
23	20.711	1.991		23	-11.274*	0.001
			21	22	14.755*	0.000
			22	23	-14.192*	0.000

Symbol name of treatment is presented at Table 2.
Standard Error of one-way descriptive and multiple comparisons LSD are equal 1.940 and 2.709, respectively.
*The mean difference is significant at the .05 level.

According to results of visual degradation grading and the least significance difference of mean percent of weight loss, the resistance (from strong to weak) of samples against Microcerotermes diversus is as follows: 1- 3-plywood, 2- particleboard and 3- medium density fiberboard (Tables 2 and 3). But based on results of mean percent of weight loss, the amount of degradation particleboard at Sistan is more than Jiroft. Also, both 3-plywood and medium density fiberboard that exposed the termites at region Jiroft and Sistan are the same degrade. The best biological resistant against the termites attack to wood-based panels is -plywood (Tables 2 and 3).

The correlation between the weight losses versus degradation grading indicated that these two properties were somehow related, though the R-square value was not very high (0.63 or 63%) (Fig. 2). This correlation was attributed to the fact that larger number of nibbles and visual penetration is indicative of higher degree of attack; consequently higher weight losses can be expected. The weight losses has shown the best actual degradation when compared to visual degradation grading because visual illusion. Termite attack continuously performed in the shelter an external thin coat. The coverture is known as carton. The wood composites under attack continuously degrade during visual observations. Since, the precision of visual degradation grading as Table 2 and Fig. 1 can be reduced by the carton, and the estimation of weight loss as Table 3 has the most validated. Generally, Table 3 shows that particleboards exposed to Microcerotermes diversus termite at Sistan region were LSD significantly different from Jiroft region. The different filed and experimental conditions can lead to such different results. On the other hand, the results of visual degradation grading and the least significance difference of mean percent of weight loss are show that the resistance (from strong to weak) of wood-based panel samples against Microcerotermes diversus is the same. It means that 3-plywood have the highest resistances against degradation of termites.

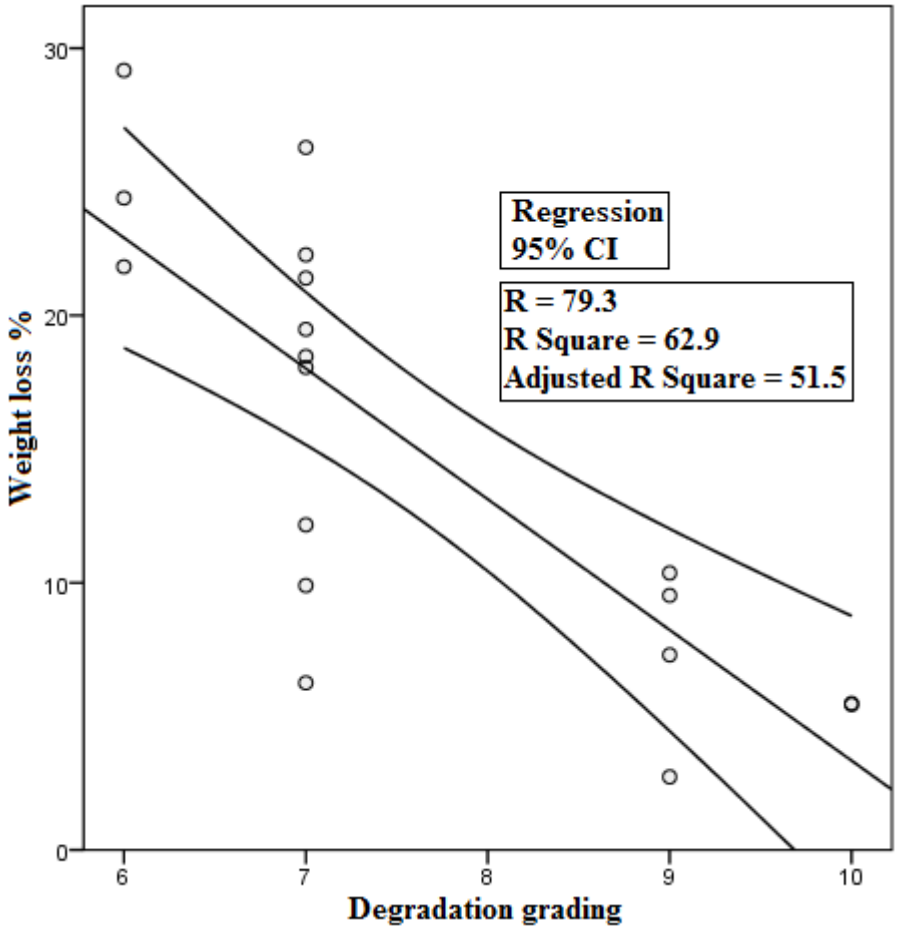

Fig. 2.
Fitted-line plot between weight losses versus degradation grading for the wood-based panels specimens.

Observation results have shown that the Microcerotermes diversus termite penetrates in cross-section. Also, the degradation of particleboard specimens exposed to Microcerotermes diversus termite at Jiroft and Sistan is significantly difference. Particleboard has the most penetrate of Microcerotermes diversus termite in cross-section when it is put at Sistan region.

The medium density fiberboard specimen has the lowest degrade especially at Jiroft (tables 3). The order of resistance against the attack of termites to wooden products is 3-plywood, particleboard and medium density fiberboard (Tables 2 and 3). Wooden composites of the 3-plywood are the most resistant samples against termites. The glue line on both sides of kernel layer prevents from attack of termites.

Published data showed that glue can be ply as composites protection against the biological degradations attack (Carll and Highley 1999; Kartal and Clausen 2001; Vick et al. 1996; Wagner et al. 1996). Before glue line hardening, the adhesive penetrate in the wood surface. The glue acts as a toxin (due to formaldehyde) against termites. The depth of glue penetration is considerable at 3-plywood because of thin layers, and it prevents of termites attack. Generally, the covered surface with adhesive reduces when component of wood-based panels is finer. Subsequently, the weighing value of adhesive increases, but glued surfaces of particles reduce. Therefore, the adhesive amount of fiberboard per unit area is less than 3-plywood and particleboard, and fiberboard is more degraded by termites. Kose et al. (2011) revealed that have not significant difference between fiberboard and particleboard against termite genus Coptotermes formosanus in laboratory. Wood-based panels consist about of 8-12% resin. Wooden products are often made from urea formaldehyde and melamine formaldehyde glues. Formaldehyde is a gas with a strong odor. It is reason that 3-plywood has greater resistance against Microcerotermes diversus termite attack compared to particleboard and MDF samples.

Wood polymer of medium density fiberboard is hydrolyzed and hemicelluloses decompose for polymerization and attachment to hydrocarbons. Also, Lignin is decomposed to finer parts that are used as composites fillers. For this reason, medium density fiberboard is a good feeding source for termites. Experimental studies indicate that medium density fiberboard is weekly act against termite degradation and it has more degraded than composites other (Kartal and Green 2003; Kose et al. 2011). The acidity of wood may increase by oxidation of the extractives and hydrolytic degradation of the wood components. (Choon and Roffael 1990; Nawawi et al. 2001). The acidity of wood is caused mainly by free acids and acidic group such as hydrolysable acetyl groups of wood. The cellulose and hemicellulose contribute to the wood acidity (Fengel and Wegener 1989). The wood acidity has an influence on the wood digestion by termite. Changes in environmental condition are causes of varied wood acidity (Sadeghifar et al. 2010).

Termite penetrates in cross-section of wood-based panels, easily. The composites thickness is covered with type laminates. Composites can be protecting with laminate. Termite penetrates through a very small hole that created at this coating. The thin skin will hide attack of termite. Medium density fiberboard is combination of wooden lingocellulosic fibers. In fact, wood chips are changed into separated fibers. Their lignin and hemicelluloses are soft and often degradable. Separation of fibers occurs in medium density fiberboard and extractive materials of fiberboard reduce in manufacture process. Cellulose is very palatable for termites. As a result, medium density fiberboard is a good feeding source for termites.

CONCLUSIONS

Studies conducted on biological resistant of productions against termites as in field condition are limited. Wood-based panels were not resistant against termites attack. The ability of Microcerotermes diversus termites to decompose medium density fiberboard, 3-plywood and particleboard by measuring weight loss and visual degradation grading evaluated. According to results of visual degradation grading and the least significance difference of mean percent of weight loss, the resistance of samples against Microcerotermes diversus is as follows: 1- 3-plywood, 2- particleboard and 3- medium density fiberboard. The 3-plywood has the highest resistance against termite attack. Observation results during 15 months in field condition have shown that the termite degradation is the same at two regions, except of particleboard specimen degradation. Adhesive such as formaldehyde can be ply as 3-plywood protection against the biological degradations attack. Separation of fibers occurs in medium density fiberboard and extractive materials of fiberboard reduce in manufacture process. There, wood components is hydrolyzed and attachment to hydrocarbons. Their lignin and hemicelluloses are soft and often degradable. Also, the wood acidity increase by oxidation of the extractives and hydrolytic degradation of the wood components. For this reason, medium density fiberboard is a good feeding source for termites.

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EFFECT OF BLEACHING CHEMICALS ON SOME VARNISHING PROPERTIES OF SPRUCE WOOD

Özlem BOZDOĞAN BALÇIK
Karadeniz Technical University, Faculty of Forestry
Department of Forest Industry Engineering, Trabzon, Turkey
Tel: 0090 462 3771527, Fax: 0090 462 3257499, E-mail: ozlembozdogan25@gmail.com

Turgay ÖZDEMİR
Karadeniz Technical University, Faculty of Forestry
Department of Forest Industry Engineering, Trabzon, Turkey
Tel: 0090 462 3771527, Fax: 0090 462 3257499, E-mail: turgay@ktu.edu.tr

Abstract

This study was performed to determine the effect of bleaching chemicals on surface adhesion strength and surface roughness in Spruce wood. For this purpose, five different bleach chemicals like sodium hydroxide-hydrogen peroxide, oxalic acid, peracetic acid, peracedic acid diluted 1/3, peracedic acid diluted 1/6 and spruce wood (Piceaorientalis L. (Link.)) were used. In this study,effect of heartwood, sapwood ratio and flat, edge grained cross section surface roughness and adhesion strength on spruce wood were determined. All specimens were varnished with cellulosic varnish. Surface roughness of coated samples was also measured using a stylus method. Adhesion strength was determined according to related standards for bleaching and control samples. The results indicated that all bleaching chemicals increased the surface roughness. The highest adhesion strength was determined for the samples treated with perasetic acid bleaching.

Key words: *spruce; bleaching; surface roughness; adhesion strength.*

INTRODUCTION

Each type of wood species has its own variation of color, texture and grain pattern. Some cuts of solid wood and flitches of veneer may be lighter or darker than others. To obtain a uniform color for use in furniture, the choice is generally limited to a color equal to or darker than the natural color of the wood. The only way to avoid this darkening is to bleach the wood or use a bleaching toner on the wood before finishing (Gerard 1983) There are two reasons for the discoloration of wood. The first is damage, drying of branches, disease etc. in alive trees (Shigo 1973). The second is oxidation, iron stains, fungi discoloration and chemical stains occurring on wood cut from trees. This kind of discoloration degrades the quality of wood material (Bauch 1999). Bleaching is moving of color pigments in the structure of wood using various bleaching chemicals and bleaching systems (Ejechi and Obuekwe 1996). While there are many materials available, the most common chemicals used as wood bleaching agents are sodium hydroxide and hydrogenperoxide (Edwin and Carter 1983).

Finishing of wood material is one of the most important processes influencing overall quality of the final product. Physical characteristics, in particular, appearance of the finished product is affected by not only the type of finish but also interaction between finish and the substrate. It is a well known fact that species, wood density and roughness of the substrate are considered major parameters to have an effect on finishing process. Wood being non-homogenous material also creates certain challenges for an ideal finished member. Sapwood and heartwood ratio within its anatomical structure would also be important element affecting interaction between the finishing material and the substrate.In certain species having extractives and other chemicals in the heartwood would create some barrier having good adherence of finish to the surface of wood substrate. Various studies investigated adhesion strength of different wood species coated using different types of finishing materials (Jaic and Zivanovic 1995, Jaic et al. 1996, Zavarin 1984, Ozdemir, Hiziroglu, Malkocoglu 2009).

In one of these past works, surface roughness of beech, spruce, fir and alder specimens were measured using a stylus type equipment before they were coated with cellulosic based varnish (Ozdemir and Hiziroglu 2009). Adhesion strength of such samples was determined and it was found that rougher specimens resulted in higher strength values than smoother samples (Ozdemir and Hiziroglu 2009). In another study, moisture content of different wood species was determined as an important factor influencing overall adhesion strength of the finish (Ozdemir et al. 2009). Zavarin found that porosity of wood can be considered as an important factor influencing adhesion strength of

finished samples (Zavarin 1984). Another past study evaluated surface characteristics of radial and tangential grain orientations of three different hardwood species and concluded that rougher surfaces required higher amount of finishing material and overall quality of finishing was influenced by the surface roughness of the substrate (Ozdemir and Hiziroglu 2007). Adhesion strength of oak and beech specimens coated with polyurethan varnishes was studied by Jaicand Zivanovic.It was found that 10.3% moisture content of the samples resulted in the highest adhesion values for both species (Burdurlu et. al. 2006). Pull-off test set up is one of the most commonly used one to determine adherence quality between finish and substrate. Adhesion strength of cellulosic varnish coated wood species as function of their surface roughness was evaluated using a pull-off type equipment by Ozdemir and Hiziroglu (Ozdemir et al. 2009).

Adhesion strength of different wood species including beech, alnus, fir, spruce, oak and maple have also been evaluated as function of their surface roughness, moisture content and type of coating materials in several studies (Jaic and Zivanovic 1995, Jaic et al. 1996, WoodHandbook 1999). Currently there is very limited information on adhesion strength of wood species coated with cellulose varnish as function of sapwood and heartwood ratio. Therefore, the objective of this work was to get an initial data on adhesion strength characteristics of such samples from four species, namely beech, alder, fir and spruce coated with cellulose varnish. Results from this work are expected to be used as quality control tool to finish these species with a better efficiency and effectively so that any furniture of cabinet members manufactured from these species can be used more efficiently during their service life.

OBJECTIVE

The main objective of the present research was to evaluate bleaching effect on the varnish properties of spruce wood. Five different bleach chemicals which sodium hydroxide-hydrogen peroxide, oxalic acid, peracetic acid, peracedic acid diluted 1/3, peracedic acid diluted 1/6 and spruce wood (Piceaorientalis L. (Link.)) were used. In this study, the effect of heartwood,sapwood ratio and flat, edge grained cross section surface roughness and adhesion sterngth on spruce wood were determined.

MATERIALS AND METHODS

The wood species, namely spruce (Picea orientalis L. Link) were used for the experiments. A total of 300 defect free heartwood (flat and edge grained) and sapwood samples (flat and edge grained)with dimensions of 400 mm by 100 mm by 200 mm were prepared and conditioned in a climate room having a relative humidity of 65 % and a temperature of 20^{o}C until they reach to equilibrium moisture content of 12%. Conditioned specimens were sanded with 80-grit and 180-grit sand paper using a commercial sanding machine (Feed speed: 12 m/min, sanding pressure: 0.5 MPa). A stylus type equipment, Mitutoyo SJ-301 profil meter was employed to measure surface roughness of the samples. Equipment has stylus with 2.5 µm radius and 90° contact angle running at a speed of 0.5 mm/s. A total of 40 random measurements with a span of 15 mm were taken from the surface of each sample in radial and tangential direction across the grain orientation. Mean peak-to-valley height (Rz) was used as an indicator for the surface quality of the samples (Wick et al. 1998, Vistosyte et al. 2012). In the next step both heartwood and sapwood specimens were coated with cellulosic based varnish using a pressurized spray gun at a spread rate of 120 g/m^2 and cured in the convection drying chamber.

Erichsen Adhesion-525 MC pull-off type tester was employed for adhesion strength evaluation of the specimens. Twenty five random measurements were taken from the surface of the samples by gluing steel head with 20 mm diameter using epoxy resin on the samples. A constant speed of 100 mm/min was applied the force to the surface layer by pulling the coating from the surface and adhesion strength value of the finishing was determined in N/mm^2 on the display of the pull-off testing unit. Five mm small cubes from each species were also cut for microscopic evaluation. Scanning electron microscope was used to determine penetration of the coating on cross section. Finally variance analysis (ANOVA) and Duncan tests were used to analyze the experimental results.

RESULTS AND DISCUSSION

The results of percenatage of surface roughness are presented in Table 1. Statistical analysis (Table 3) showed that there was a significant effect between five different bleach chemicals [sodium hydroxide-hydrogen peroxide, oxalic acid, peracetic acid, peracedic acid diluted 1/3, peracedic acid diluted 1/6 on the spruce wood. This study effect of heartwood, sapwood ratio and flat, edge grained cross section surface roughness on spruce wood were determined (Fig. 1). As it is expected surface roughness is usually considered as intent physical property of wood and wood based products.

According to Table 1, spruce edge grained sapwood which was pretreated with peracedic acid diluted 1/6 had the highest surface roughness value with 57.533. The spruce flat grained sapwood which was pretreated with oxalic acid had the highest surface roughness value with 69.954. The spruce edge grained heartwood which was pretreated with peracedic acid diluted 1/3 had the highest surface roughness value with 132.560. The spruce flat grained heartwood which was pretreated with sodium hydroxide-hydrogen peroxide had the highest surface roughness value with 109.610.

Table 1

Results of percentage of surface roughness

		2	3	4	5	6
Sapwood	**EdgeGrained**	20.286 (3.057)	54.674 (9.681)	31.136 (3.213)	51.777 (5.769)	57.533 (12.310)
	Flatgrained	36.920 (8.632)	69.954 (14.613)	60.834 (7.207)	56.812 (7.787)	56.367 (16.449)
Heartwood	**EdgeGrained**	103.550 (22.759)	78.502 (15.780)	108.620 (26.786)	132.560 (36.999)	51.355 (12.715)
	Flatgrained	109.610 (35.904)	70.825 (11.747)	44.473 (9.050)	66.623 (25.299)	68.832 (17.359)

2-sodium hydroxide-hydrogen peroxide,3-oxalic acid, 4-peracetic acid, 5-peracedic acid diluted 1/3, ,6-peracedic acid diluted 1/6

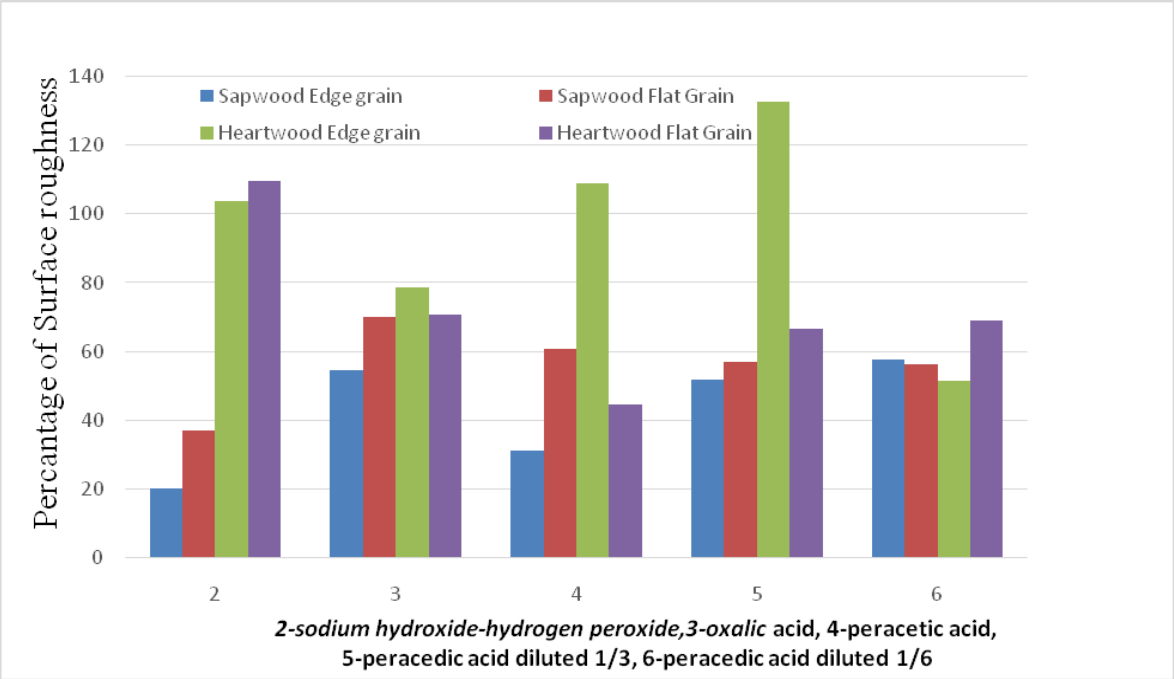

Fig. 1.
Percentage of surface roughness.

As a results in Table 3, there was no significant difference between flatgrained and edgegrained surface rouhghness. On the other hand, there was a significant diference between bleaching chemicals in Table 3. As can be seen Table 4, peracedic acid diluted 1/3 had higher surface roughness than the other bleaching chemicals.

Table 2

Results of adhesion strength (N/mm^2)

SPRUCE				
Sapwood/Heartwood	Edgegrain/flatgrain	NO*	Average	Standard Deviation
Sapwood	EdgeGrain	1	1,49	0,37
		2	1,59	0,08
		3	0,82	0,35
		4	1,84	0,08
		5	1,80	0,11
		6	1,76	0,15
		Toplam	1,55	0,41
	FlatGrain	1	1,56	0,13
		2	1,68	0,17
		3	0,98	0,26
		4	1,80	0,16
		5	1,78	0,19
		6	1,73	0,16
		Toplam	1,59	0,33
Heartwood	EdgeGrain	1	1,55	0,20
		2	1,75	0,14
		3	0,86	0,30
		4	1,75	0,10
		5	1,76	0,19
		6	1,72	0,14
		Toplam	1,57	0,37
	Flatgrain	1	1,63	0,13
		2	1,76	0,14
		3	0,74	0,20
		4	1,80	0,08
		5	1,78	0,15
		6	1,79	0,15
		Toplam	1,58	0,41
*1:Control, 2-sodium hydroxide-hydrogen peroxide,3-oxalic acid, 4-peracetic acid, 5-peracedic acid diluted 1/3, ,6-peracedic acid diluted 1/6				

According to Table 2, all bleaching chemicals increased the adhesion strength. The highest adhesion strength was determined for peracetic acid bleaching. Spruce edge grained sapwood which was pretreated with peracetic acid had the highest adhesion strength with 1.84 N/mm^2. According to Table 3, effect of heartwood and softwood and effect of edge grained and flat grained were not significant. but there was a significant difference between bleaching chemicals. While peracetic acid had the highest adhesion strength, oxalic acid had the lowest adhesion strength.

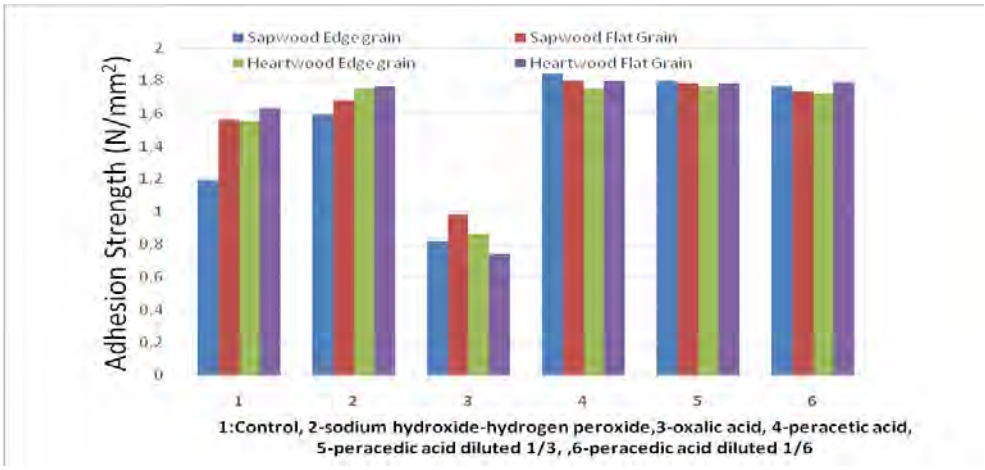

Fig. 2.
Adhesion strength.

Table 3

Statistical analysis of the roughness and adhesion strength -test results

Surface Roughness					
Source	Sum of squares	df	Mean square	F value	Significance level
Effect of heartwood and sapwood (A)	34406,601	1	34406,601	106,938	***
Effect of flat and edge grained(B)	712,556	1	712,556	2,215	NS
Effect of bleaching chemicals(C)	4925,570	4	1231,393	3,827	**
AxB	9687,415	1	9687,415	30,109	***
AxC	20967,353	4	5241,838	16,292	***
BxC	7888,633	4	1972,158	6,130	***
AxBxC	12556,345	4	3139,086	9,756	***
Error	32174,466	100	321,745		
Total	654982,486	120			
Adhesion Strength					
Source	Sum of squares	df	Mean square	F value	Significance level
Effect of heartwood and sapwood (A)	0,002	1	0,002	0,052	NS
Effect of flat and edge grained(B)	[illegible]	1	[illegible]	[illegible]	NS
Effect of bleaching chemicals(C)	26,472	5	5,294	144,037	***
AxB	0,009	1	0,009	0,242	NS
AxC	0,308	5	0,062	1,678	NS
BxC	0,4	5	0,008	0,217	NS
AxBxC	0,252	5	0,5	1,37	NS
Error	7,94	216	0,37		NS
Total	630,923	240			NS

N.S: Non-significant *Significant at the α=0.05 level **Significant at the 0.01 level *** Significant at the α=0.001 level

Table 4

Duncan test results

Strength Properties	**Factors**	**LS Mean**	**Homogenous Groups***
Percentage of Surface Roughness	Bleaching Chemicals		
	sodiumhydroxide-hydrogen peroxide	7.593	ab
	oxalic acid	68.488	ab
	peracetic acid	61.265	a
	peracedic acid diluted 1/3	76.942	b
	peracedic acid diluted 1/6	58.521	a
	Grain		
	Edge grain	68.999	a
	Flat Grain	64.125	a
	Wood		
	Heartwood	83,495	a
	Sapwood	49,626	b
Adhesion Strength	Bleaching Chemicals		
	Control	1,56	b
	sodiumhydroxide-hydrogen peroxide	1,7	c
	oxalic acid	0,85	a
	peracetic acid	1,8	d
	peracedic acid diluted 1/3	1,78	cd
	peracedic acid diluted 1/6	1,75	cd
	Grain		
	Edge grain	1,527	a
	Flat Grain	1,555	a
	Wood		
	Heartwood	1.544	a
	Sapwood	1.538	a

CONCLUSIONS

The effect of bleaching chemicals on the spruce wood quality was evaluated. Bleaching chemicals increased surface activation. Thus, the results indicated that, all bleaching chemicals increased the surface roughness. The highest adhesion strength was determined for peracetic acid bleaching.

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SECTION 6.

CONSERCATION-RESTORATION OF WOODEN OBJECTS

IN SEARCH OF A SUITABLE COATING FOR PYROGRAPHY

Susan M. MILLIS
Private artist, conservator and researcher in pyrography
Address: The Flat, 345A Desborough Avenue, High Wycombe HP11 2TH, UK
E-mail: smmillis@hotmail.com

Abstract

This paper presents the results of an experiment made to examine the discolouration of pyrography, exposed to natural light aging, after coating it with one of two traditional surface finishes. Strips of wood material made of English sycamore (Acer pseudoplatanus) were 'scorched' in a controlled manner at various temperatures to produce different shades from light to dark. This method produced consistent and comparable colour scales for the purpose of testing. Samples were then coated with an oil solution, shellac, or left uncoated and exposed to natural light aging for 110 days/nights. Overall, the sample coated with the oil solution returned the lowest colorimetric colour differences for segments 'scorched' at 350°C and above. Yet, from a visual perspective, via microscopy, it was found that the use of either coating was beneficial for the longevity of the surface 'scorching', in comparison to the sample left uncoated. Neither coating seemed to retard fading for pyrography 'scorched' at lower temperatures. However, it must be considered that the use of either of these treatments will eventually reduce contrast in the image as the coatings darken and yellow further in natural light.

Key words: *pyrography; surface coatings; colour change; oil; shellac.*

INTRODUCTION

Pyrography and poker work are largely considered to be the generic names for a collection of techniques, used to make pictures and designs by 'scorching' a receptive surface to various shades of brown with a hot tool. Although objects can be found made of other organic materials in museum collections, such as leather, paper and velvet (Millis 2004), this paper deals with pyrography when applied to a wood substrate. Wood was clearly the most popular support for this work throughout history, and it is still much favoured today. Fig. 1(a) pictures a pyrography panel by Joseph Smith, c. 1820. Thought to be made of sycamore, it is after an original painting by Willem Drost (1633-1659) dated 1654. However, this panel might well have been worked from the mezzotint engraving published by William Pether (c. 1738-1821) on 1 August 1768, which is also shown in Fig. 1(b). Pether made engravings of a number of paintings, attributed at the time to Rembrandt van Rijn (1606-1669). Until the end of the eighteenth century, the Drost painting was attributed to Giorgione (d. 1510) (Bikker 2005).

a

b

Fig. 1
(a) 'Portrait of a Man with a Plumed Beret', by Smith c. 1820. Photograph by Susan M Millis (b) The mezzotint print, by William Pether, 1768, AN841924001 ©The British Museum

Contrary, perhaps, to popular belief, in a previous article (Millis 2013) it was shown that uncoated decoration made by pyrographic methods is subject to significant fading when exposed to direct solar radiation, which has the potential to dramatically reduce the aesthetic appeal in a few short months if left unchecked. This is also dependent on the species, density, cut, and moisture content, of wood selected as a support, and the temperature at which it was made. There are certain factors governing how the pyrographic image might be seen to deteriorate. Perhaps, one of the main drawbacks is caused by the way a light-coloured wood surface oxidises on exposure to light, producing yellow-coloured deterioration products in the lignin fraction, particularly. Many of these are thought to be ortho and para quinonoid structures (Feller 1971, Hon 1991). For further explanation of this issue, it is important to note that at a microscopic level, and especially when made at low temperatures, pyrography does not affect all of the surface molecules to the same degree, as can be seen in Fig. 2. This is because of the difference in thermal reactivity between molecules. Therefore, as illustrated by the arrows in the image, there are still areas that can be oxidised by light in the expected way. The question here is can light-coloured pyrography be thought to be fading where, in truth, the development of these structures in the molecules unaffected by heat is effectively obscuring it?

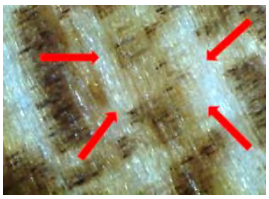

Fig. 2.
A lightly 'scorched' surface at x400 magnification.

Further to this, carbonyl groups are often a main feature in chains made of conjugated double bonds and are generally considered to be the main chromophoric group found in the discolouration of some traditional surface coatings as well. As they form more and more conjugated double bonds, the colour of these chromophores becomes stronger, thus absorbing energy at increasingly longer wavelengths in the visible spectrum rather than the ultraviolet. For further clarity of this point, Feller (1971) draws the reader's attention to acetone, which is colourless CH_3COCH_3, diacetyl, which is yellow $CH_3COCOCH_3$, and triketopentane, which is yellow-orange $CH_3COCOCOCH_3$. This example shows how the addition of just one carbonyl group in a conjugated chain can cause the molecule to respond to a longer wavelength. Thus, pyrography 'scorched' at low temperature settings and protected with a surface treatment, might also be disguised by this discolouration occurring in the coating. This will leave the pyrographic artist contending with not only that their work fades but also that parts of the wood yellow simultaneously and if the work is coated, changes occurring in the coating could potentially render their work almost indistinguishable, in a short space of time.

Surface Coating

The application of a surface coating to any artwork is a very personal decision. Much is dependent on how the artist visualizes the final object. For example, for a dark pyrographic, framed work, where most of the surface is 'scorched', there would be little need to retard the photo oxidation of the bare wood. Therefore, there would be several finishing options for the artist to consider. Yet, in the current author's case, the objects are free standing so any coating application needs to withstand regular handling. Recommendations, from practitioners, can be found in the literature for incorporating resins derived from polyurethane, alkyl and acrylic resins (Poole 1995), as well as wax paste and varnishes containing UV inhibitors (Irish 2010). First, however, the overall question must be answered, to finish or not to finish?

OBJECTIVES

To help make that choice, this paper investigates the application of two simple surface coatings, to see if they offer any benefit for the longevity of pyrography artwork, during exposure to direct solar radiation filtered through window glass. Research examining the light resistance of coated samples has not been published previously in academic circles. Therefore, this study offers a contribution to the available knowledge of pyrographic surfaces.

METHOD, MATERIALS AND EQUIPMENT

Wood samples

The term 'samples' in this case, refers to strips of wood material supporting surface colour change as a direct result of heating with a hot tool. Samples made from English sycamore (*Acer pseudoplatanus*) were examined in this project.

The sycamore wood was sawn into sheets averaging in size 405 mm x 205 mm x 1.2 mm (longitudinal by radial by tangential). They were abraded with P320 carbide paper, followed by 00 'flour' glass paper, to a smooth surface. Assessment was made by touch. Moisture content of 8.4% was determined using the oven dry method as defined by BS EN 13183-1:2002, and density calculated at 631kg/m^3, based on the oven dried material.

Three wood strips per sheet were 'scorched' at a range of temperatures from 200^{o}C to 450^{o}C with incremental changes of 25^{o}C. The 'scorching' method was the process used in previous work that utilised a temperature controlled stylus, which was driven from side to side in a smooth and linear fashion at a uniform speed, leaving an impression on the surface. Each temperature segment was 40 mm wide and, a minimum of 20 mm deep. This method produced consistent and comparable gradient scales for the various temperatures and one untreated section to act as a control. All samples were stored in the testing environment to equilibrate with the conditions for 14 days. The scales were then individually cut from the sheets and prepared for use (Millis 2013).

Guides to coating

The coating solutions considered in this research were traditional; linseed oil and shellac. These were chosen to represent the type of wood finish likely to have been used on pyrographic artefacts in the past, for protection.

Shellac

The links with shellac are grounded in furniture history and conservation. Lewis (1979) and Hahn (1994) both advised using it to protect objects of historical American pyrography. The panel shown in Fig. 1(a) was originally coated with a plant resin. However, UVA fluorescence also detected a quantity of shellac covering the central area, possibly put into place during a past conservation treatment. When the coatings were removed it became clear that it had given the piece fair protection, as the very gentle shading forming the facial details was still detectable with the naked eye. Previously, this shading had been concealed by the coatings.

Advantages

Shellac is relatively easy to apply. It is available in different grades and colours. It should remain reversible, though time promotes a gradual reduction in this property (Rivers and Umney 2003).

Disadvantages

Shellac discolours with light exposure, which will enhance the effect of yellowing observed in the plain surface of light-coloured wood. Upon removal, the polar solvents used will affect the pyrographic image, causing some inevitable colour loss (Millis 2012).

Linseed oil

The use of an oil to coat contemporary pyrography, was recommended to the current author by a colleague (Muradian 2004), as a successful method of protection, which prompted the original study. Furthermore, Walsh (1992) clearly encouraged her readers to use furniture oil to clean and protect historic objects of Australian pokerwork. So, there is evidence to show that others have believed oil to be a suitable coating for pyrography. In the experiment described here, linseed oil was selected because of the strong association that it has in both furniture and easel painting history. However, from a conservation perspective, oil would not usually be considered appropriate for coating works of art.

Advantages

Oil is easy to apply, and miscible with aliphatic and aromatic solvents such as toluene and white spirit. It will saturate the overall colours of the work. It does not appear to cause harm to the pyrographic surface (Millis 2012).

Disadvantages

Linseed oil becomes increasingly darker with light exposure and cross-links severely. It will strongly colour the untouched parts of the image, thus reducing contrast. It is most likely to become

intractable from the surface (Williams 2003), or need polar solvents (Rivers and Umney 2003), perhaps dichloromethane (Sawyer 2017), to remove it. These will damage the surface. Winter (1983) noted that carbon pigments retard the free radical drying mechanism observed in the oil vehicle. This was considered to be a quenching reaction caused by free radicals in the carbon pairing with those in the oil. Therefore, as carbonaceous radicals are likely to be involved in the pyrographic surface, the image may become tacky and pick up particulate matter.

Manufacture and application of coatings

The two traditional surface coatings were made from raw materials.

Shellac

100 g de-waxed blonde shellac (ground), in 300 ml Industrial Methylated Spirits (IMS), VWR, GPR, 98/99% w/w total alcohols. A coating of shellac varnish was applied to two of the 'scorched' samples by brush, followed by a second coating an hour later.

Linseed oil

100 ml of boiled linseed oil, in 100 ml of Stoddard Solvent, a white spirit/hydrocarbon solvent, VWR, GPR. An initial coating of the oil solution was applied to two of the 'scorched' samples by brush, followed by a second coating an hour later.

Two samples were left uncoated.

Fig, 3, shows part of a sample scale made of sycamore, which was coated with shellac. It was soon to be realised that segments 'scorched' at less than 325^{o}C were completely reversed in the test, and there were only small differences in colour between those 'scorched' at the highest temperatures. Therefore, as an aid to clarity in the results presented here, they are not mentioned further. In this image, each segment of interest is marked with the temperature of execution, showing clearly the influence that heat has on the colour of wood. The segment marked with brackets is shown here for colour information only.

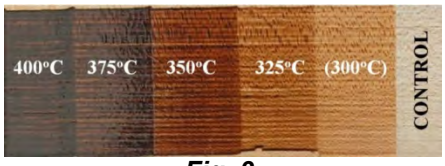

Fig. 3.
A colour scale produced of English sycamore, coated with shellac, showing the effect of heating on wood colour.

Three colour scales, one uncoated, one shellac coated and one oil coated, were stored in complete darkness until the end of the testing process, to act as overall control samples.

Natural light aging

The purpose of this test was to replicate the effects of light exposure on pyrographic decoration in a usual interior situation. To that end, the term natural light aging refers to exposing the samples together day and night, through window glass, on a south-westerly facing window sill for 110 days/nights. At location 51.680, -0.802, and an altitude of 204.0 m above mean sea level. Exposure took place between June and October.

Why use natural light?

Whereas it might be thought that using an accelerated aging process would produce faster and more reproducible data, it is largely dependent upon the light source available. Searle (1994) states quite clearly that…'the type of light source used in durability testing significantly influences the stability ranking of materials as well as the mechanisms and type of degradation'. Many tests were conducted, during the overall research project, under a metal-halide UVA lamp (https://www.hoenlegroup.com). However, the results were not comparable to those produced in natural light.

The display was based on British Standards Institute BS EN ISO 105-B01:1999 and set up as follows: The standards were met as closely as possible. The wood sample colour scales were divided into three even-width vertical strips with a pencil and mounted together on foam core board by covering the centre section with a strip of card, lined with MT20 ultraviolet protective film (http://www.sun-x.co.uk/products/mt20-dark-neutral-uv-window-film), which was secured in place over each sample with two brass tacks (Fig. 4, Cover A). The left and right thirds of each sample were

exposed to natural light for 50 days/nights. Then the right third was covered with lined card in the same way (Cover B) and the left third was exposed for an extra 60 days/nights. Therefore, for each sample, the right third was exposed for 50 days/nights, the left third was exposed for 110 days/nights and the centre third was not exposed to light (Millis 2013).

Light exposure for the 110 day period was quantified in lux as 10,326,000, by monitoring an in situ LightCheck® dosimeter (http://keepsafe.ca/LightCheckHome.htm), and was based on the results of four trials. No attempt was made to measure UV radiation, and the control of temperature and relative humidity were beyond the scope of the study (Millis 2013).

Colour measurement

As the colour of pyrography is heterogeneous, for accurate colour comparison to be made, it was essential to make sure that the colorimeter was sited at the same place each time a reading was taken. This was because of the influence wood grain has on the surface colour. To aid in this, a template was made from Perspex. It consisted of a shallow tray with built-up sides and was 45 mm wide in the centre, just large enough to insert the sample. Another piece of Perspex fitted closely inside, into which three 22 mm holes were bored; one to the left, another to the right and a third in a central position. These allowed enough room for the tip of the colorimeter to be sited flush with the sample. Paper rulers were adhered to each rim of the template, which permitted pin-point accuracy to be observed. Only readings made in the same position of the same sample were compared (Millis 2013).

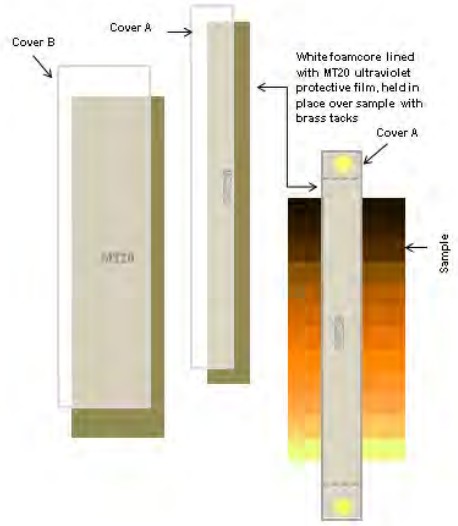

Fig. 4.
Natural light aging, sample preparation plan (Millis 2012, Millis 2013).

A Konica Minolta Chroma Meter CR-300 was used to monitor changes in the surface colour of the 'scorched' samples. The measuring head of the instrument incorporated an 8 mm measuring area, was index set to use D65 illumination and calibrated to a 2^{o} observer angle. Calibration was performed at the start of each measuring session. Measurements were taken from the left side, right side and the centre section, making a total of 36 readings for each sample scale, covering the full twelve segments of colour change. The colorimeter, fitted with a 22 mm light protection tube (CR-A33a), was index set to take three tristimulus measurements and then calculate a mathematical average for the segment. The *CIE L*a* b** (1976) colour space was selected for interpretation. For this system *L** represents lightness and is on a scale of 100, where *L** = 100 is white and *L** = 0 is black. The *a** measurement characterises the green (- *a**) red (+ *a**) axis and *b** the blue (- *b**) yellow (+ *b**) axis. All measurements taken were absolute. Total colour change was calculated from these measurements by using the following equation (1) (for full method see BS EN ISO 105-J03:1997):

$$\Delta E^*_{ab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

$$Where \quad \Delta L^* = L^*_T - L^*_R$$

$$\Delta a^* = a^*_T - a^*_R$$

$$\Delta b^* = b^*_T - b^*_R$$

R = Reference sample (before exposure)

T = Test sample (after exposure)

Before exposure began the colour parameters were determined for each segment, with relevant examples presented in Table 1(a). Subsequent measurements were mathematically compared to these data sets in order to gain an accurate insight to the photochemical stability of the pyrographic image after surface coating.

RESULTS AND DISCUSSION

The colour changes that took place in the samples were the direct result of exposure to natural light, through window glass, for 110 days and nights. They are presented here in chart form with the accompanying computational colour differences shown in Table 1(b). In these charts, 0 represents no change in colour at all. A difference of just *CIEΔE*$^*_{ab}$ 3 has been determined to be the minimum value of colour change that can be recognised by the human eye (Hon and Minemura 1991, Sundqvist 2004, Millis 2013). The first chart, pictured in Fig. 5, represents the absolute colour differences caused by exposure, at the end of the testing phase (ΔE^*_{ab}).

Table 1

Colour parameters of selected samples before exposure to natural light, with colour difference values recorded after exposure (grey)

Sycamore	Temperature	(a) Colour measurements before exposure			(b) Colour differences after exposure			
		L^*	a^*	b^*	ΔE^*_{ab}	ΔL^*	Δa^*	Δb^*
Uncoated	**Control**	**78.43**	**6.70**	**16.78**	**11.94**	**-1.91**	**0.09**	**11.79**
	325°C	**52.20**	**11.88**	**20.79**	**21.52**	**18.07**	**-2.09**	**11.05**
	350°C	**39.71**	**10.80**	**13.63**	**24.49**	**17.68**	**2.03**	**16.83**
	375°C	**35.73**	**7.72**	**8.56**	**20.64**	**13.07**	**4.37**	**15.36**
	400°C	**35.72**	**5.97**	**6.11**	**19.34**	**11.94**	**4.68**	**14.47**
Shellac	**Control**	**73.78**	**7.59**	**22.48**	**9.28**	**1.25**	**0.26**	**9.19**
	325°C	**50.43**	**12.62**	**24.48**	**22.53**	**19.54**	**-2.11**	**11.02**
	350°C	**36.81**	**12.22**	**16.28**	**27.39**	**20.42**	**1.69**	**18.17**
	375°C	**30.83**	**9.27**	**9.23**	**24.35**	**15.02**	**5.53**	**18.35**
	400°C	**28.81**	**6.68**	**5.62**	**19.94**	**11.78**	**6.15**	**14.87**
Oil	**Control**	**75.81**	**8.90**	**26.57**	**9.90**	**-3.18**	**1.7**	**9.22**
	325°C	**39.56**	**13.30**	**17.53**	**22.33**	**16.30**	**0.74**	**15.24**
	350°C	**31.78**	**9.22**	**8.40**	**19.14**	**11.39**	**5.09**	**14.51**
	375°C	**28.24**	**4.42**	**2.93**	**14.21**	**7.31**	**6.20**	**10.49**
	400°C	**27.94**	**1.88**	**0.73**	**9.25**	**3.76**	**5.43**	**6.47**

On first examination, it is clear that significant colour changes occurred in all samples. Starting with the control segments (not treated with heat), it is evident that the sycamore wood experienced colour change directly, showing an overall colour change value (ΔE^*_{ab}) of 11.94 for the uncoated sample, 9.28 for the shellac coated sample, and 9.90 for the oil coated sample. There is visual evidence to show that most of this occurred by a marked increase in yellowing, for all three samples, which is supported by the data presented in Table 1(b) (Δb^*). Yet, it is also important to point out that the addition of the coatings had changed the optical properties of the samples before exposure began, increasing the yellowness (+b^*) by 34% (shellac) and 58% (oil), while there were also differences in the L^* and a^* axes. This can be seen in the 'opening' data, Table 1(a).

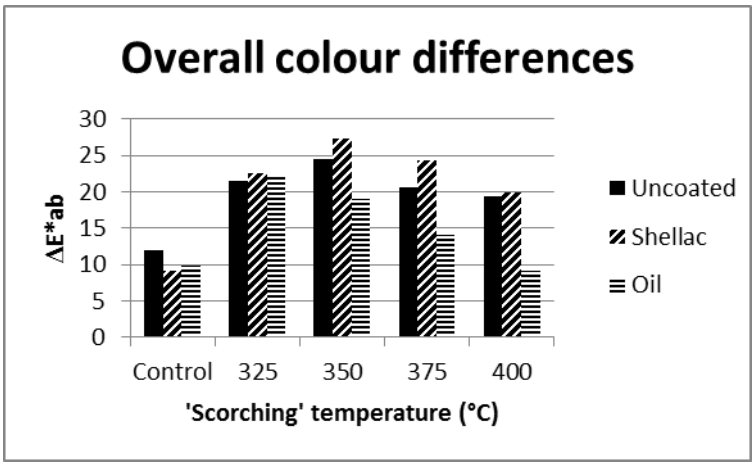

Fig. 5.
Overall colour differences for the samples, at the end of the testing phase.

For the segments 'scorched' at 325°C, marked fading was observed for all samples, which amounted to a rise of 35% of the original lightness for the uncoated sample, 39% for the shellac coated sample and 41% for the oil coated sample. Changes in lightness peaked for the segments 'scorched' at 350°C, increasing to 45% (uncoated) and 55% (shellac coated), whereas it dropped to 35% for the oil coated sample. These results are shown in Fig. 6, which maps the changes that occurred in the lightness parameters (*ΔL**). This trend continued, with the shellac coated sample providing the largest colour differences throughout. However, colour change dropped notably, for the sample coated with the linseed oil solution, returning an overall ΔE^*_{ab} of 9.25 for the segment 'scorched' at 400°C, compared with19.34 (uncoated) and 19.94 (shellac coated). This result points to an increase in performance for the oil solution used to protect the surface colour.

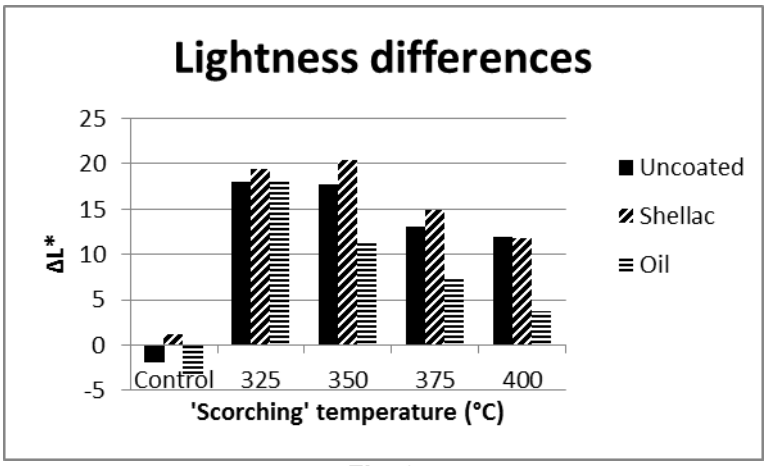

Fig. 6.
Overall lightness differences for the samples, at the end of the testing phase.

Experimental study has demonstrated to the author that, when choosing a surface coating, a completely clear, 'water-white', non-yellowing varnish, or lacquer, is unlikely to be able to provide adequate protection for fugitive (unstable) colouring matter. The UV transmission of glass is affected by the source and the age of the glass. However, it is important to remember that UV rays in excess of 310 nm can penetrate it and cause serious, long-term, deterioration to organic and light sensitive materials (Ketola and Robbins 1994). Whereas, UV shielding can be provided by incorporating ultraviolet absorbers, hindered amine light stabilisers, and inorganic nanoparticles, or by glazing with conservation glass or film, from an aesthetic point of view, these will mainly help to prevent a light-coloured wood surface from yellowing. Though, it must be acknowledged that much successful research has been achieved with them, when used to protect a natural wood surface (for a review of the research, see Evans *et. al*. 2015). However, according to Passauer *et. al.* (2015), these additives are often inadequate to afford reasonable protection to dark-coloured wood surfaces, and in the case of thermally modified timber (TMT), they have been known to accelerate discolouration when compared to unprotected samples. In an earlier paper (Millis 2013), it was elucidated that pyrography

is likely to be the result of several processes, developed between migrating extractive substances and the coloured reaction products from the thermal degradation of wood components, which combine in caramelisation and Maillard reactions, thus, forming a crust-like substance on the surface. Consequently, this crust will contain a numerous diversity of labile molecules at various stages of charring and carbonisation (Shafizadeh 1984). These molecules will absorb light strongly. Certainly, it could be speculated that, if products such as furfural or hydroxymethylfurfural are present in the pyrographic image, the use of inorganic nanoparticles particularly, in a coating, might well cause a photocatalytic reaction to occur (Ghasemi *et. al.* 2016), ultimately advancing the fading of the image. However, no research with nanoparticles has been achieved by the current author to date. To offer a good level of protection to a substrate from the UVA wavelength range (315-400 nm), any ultraviolet absorber would need to have a bathochromic shift and, thus, would impart a yellow cast to the coating. Yet, even this will not protect the surface from the other, most dangerous, wavelength, which according to Searle (1994) is at 405 nm, in the violet region of visible light. This is the optimum wavelength for bleaching of yellow chromophores to occur. Moreover, Kataoka *et. al.* (2007), determined that the blue region (434-496 nm) of visible light is also capable of penetrating wood and causing bleaching effects of the surface molecules, though, in their tests it was not found responsible for the photodegradation of the underlying layers.

Fig. 7.
The three samples at the end of the testing phase (top) uncoated (centre) shellac coated (below) oil coated.

The three samples are presented together in Fig. 7, where it is clear to see that all of the identified segments have faded. However, though a subjective determination, the yellowing that occurred in the natural wood was darker and more chromatic for the coated samples, particularly for the one treated with the oil solution, at the bottom of the picture. To investigate further, photomicrographs were taken at x400 magnification, of the exposed segments 'scorched' at 400^{o}C, after the coated samples had been gently solvent cleaned, so as not to abrade and remove the surface. These are presented in Fig. 8. Picture (a) represents the uncoated sample, picture (b) the shellac coated sample, and picture (c) the oil coated sample. Examination of these images shows that the surface of both coated samples appeared darker and more saturated than the uncoated sample, which had greyed throughout. This greying is thought to have been caused by the chemical destruction of the carbonaceous aggregates by electromagnetic radiation.

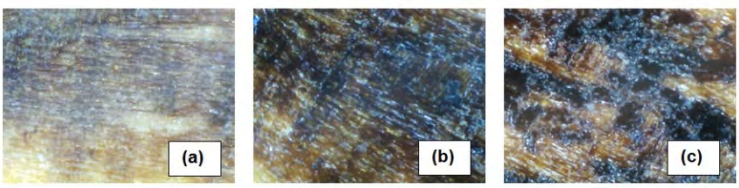

Fig. 8.
The segments 'scorched' at 400^{o}C, at x400 magnification, after exposure (a) uncoated (b) shellac coated (c) oil coated.

Though, the colorimetric data sets for the shellac coated sample did not support it, from a visual perspective, it was determined that both of the surface coatings tested gave some degree of protection to the pyrography, where 'scorched' at temperatures above 350^{o}C. Though, little shielding

was apparent for pyrography made at lower temperature settings. While clear, both surface coatings imparted a yellow hue to the samples when applied, and it could be speculated that, as such, the coatings were affording their respective surfaces protection by absorbing the blue region themselves and, thus, reflecting yellow. However, there was no doubt at all that the addition of either coating would eventually reduce contrast in the image by darkening and yellowing further in natural light.

Table 2, presents the overall colour differences seen in the control samples, which were stored in complete darkness for the duration of the testing phase. Examination of the data revealed that dark reactions occurred in both coated samples. However, for the oil coated control segment 5.49 was the difference in the *b** axis, and for the shellac coated control segment, the difference was 2.26. These results determined that the coatings had also yellowed in the dark. Though, it did not seem to affect the pyrography. There is a need for much further work to be achieved in this area.

Table 2

Overall colour differences seen in the control samples at the end of the testing phase

Segment Temperature	Uncoated		Shellac coated		Oil coated	
	ΔE^*_{ab}	Δb^*	ΔE^*_{ab}	Δb^*	ΔE^*_{ab}	Δb^*
Control	1.14	0.78	2.53	2.26	5.74	5.49
325°C	1.33	1.15	3.39	2.86	3.68	3.05
350°C	1.58	1.20	1.76	1.32	1.91	1.37
375°C	1.56	1.06	1.50	1.17	1.50	1.06
400°C	0.53	0.05	1.46	0.80	0.33	0.15

CONCLUSION

The discolouration of pyrography samples applied to English sycamore, and coated with linseed oil and shellac, has been investigated and compared to an uncoated sample, after exposing them to natural light aging for 110 days/nights. Out of the three samples, the linseed oil coated sample returned a consistently lower colour change value, and lower increases in lightness, where 'scorched' at temperatures of 350°C and above. Even though, from a conservation point of view, the use of drying oils would not be recommended, it was determined that linseed oil offered the best protection to pyrography, made at those temperature settings. However, from a visual perspective, the microscopic surface of the shellac coated sample also seemed to be in a superior condition than the uncoated sample, which appeared grey by comparison. This would indicate that shellac also offered some degree of benefit to pyrography, though, in time, both coatings would significantly reduce contrast by contributing further to the darkening and yellowing occurring in the plain wood surface. Neither coating was found to reduce the discolouration occurring in pyrography made at lower temperature settings. It was recognised that there is a need for much further research to be achieved, in the search for the most suitable coating.

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RESTORATION OF GILDED FRAMES - CHALLENGE AND SATISFACTION. A CASE STUDY

Emanuela BELDEAN
Lecturer – Transilvania University of Brasov- Faculty of Wood Engineering
Address: B-dul Eroilor nr. 29, 50036 Brasov, Romania
E-mail: ebeldean@unitbv.ro

Levente MAJOS
Eng. – TRANSILVANIA University of Brasov – Faculty of Wood Engineering
Address: B-dul Eroilor nr. 29, 50036 Brasov, Romania
E-mail: majos@yahoo.com

Abstract

The paper presents a case study of restoration of a gilded frame from the beginning of the XXth century. The frame presented a serious damage of the ornaments (gildings) that are missing, which made difficult their reconstruction. A corner was visible eroded and the joints were fragile. A visible biological attack was observed and nails interventions. The initial destination as a mirror frame was changed into a photo frame. The restoration was a challenging, very delicate and difficult work including: consolidation of the flaking gesso and gilding, cleaning, curative treatment, structural consolidation and gluing, replicating missing decorative elements, refinishing. After restoration the result was remarkable and the frame was displayed at the Faculty of Wood Engineering within the Restoration 2016 and the Researchers's Night 2016 exhibitions.

Key words: *wood; restoration; gilded frame; gesso; conservation.*

INTRODUCTION

Wooden frames have been recognised, in recent decades, as work of value that should be conserved to the same standard as other cultural material. Frame conservation has its roots in the traditional crafts of frame making and repair, shaped by modern conservation ethics and methodology. Frame conservation is part of the broader conservation field of gilded, polychrome and wooden objects conservation, and has close links to furniture conservation (McGowan-Jackson 2008). The concern for frame damages that occur during exhibitions, storage, travel, handling, adverse environment has inspired the development of the field of frames conservation, and in 1996 was established the Gilded Objects Conservation Special Interest Group of the AICCM, Australia. In Europe the concern for frame conservation lead to organisation of different meetings, presentations and exhibitions about history, materials and conservations of frames. An example is the conference: *Frames: The Northern European Tradition* (2005) held in Dresden at the Gemäldegalerie and organised by Lisa Koenigsberg through her organization Initiatives in Art and Culture, in association with New York University. In 2008, the symposium *Frames: past, present and future* has been held in Australia (McGowan-Jackson 2008). Another preoccupation in this field were pointed out in United Stated by Susan Jackson, the founder and owner of Harvard Art's, a Professional Associate of the American Institute for Conservation of Historic and Artistic Works that serve as a board member of the New England Conservation Association and the Society of Gilders. http://harvardart.com/about-us. Every museum of the world has in the conservation departments a section for wood restoration including different types of frames (simple, profiled, carved, gilded, gold leaf coated or other variety of coatings). In Chicago the Frame and Gilding Department from the Conservation Centre, the largest and most comprehensive private art conservation laboratory in the USA, specialises in the preservation and restoration of frames and objects with gold, silver, and metal leaf applied on the surface. The Center's conservators focus on objects that include period frames, gilded antiques, and furniture (http://www.theconservationcenter.com/frames-gilding). The concern for frame restoration came toghether with pictures restoration. At the National Gallery of Art (USA), the Frame Conservation Department studied the techniques and materials used on period frames (Ravenel 1994). The nomenclature and the development of popular styles and constructions of the XIXth century frames in America were presented by Glover (2006). Publications providing technical data on individual frames, including materials and methods of construction and profile drawings, are particularly useful (Thorn 1987, Glover 2006, Lizun 2012, Sandu et al. 2014, Bjorneberg 2017, Reynolds 2017). In the UK, at The Institute of Conservation, the Gilding and Decorative Surfaces Group is a forum for conservators

of a wide range of objects, from wood carving to stuccowork (https://icon.org.uk/groups/gilding-decorative-surfaces/committee).

During the history the frame evolved, from Egyptians (http://www.frameusa.com/pages/early-history-of-picture-frames), to wood multi-panel paintings from 1200s-1300s and to the Renaissance when the frame became a separate entity apart from a painting http://lauramorelli.com/history-of-picture-frames/.

The role of the frame in the presentation of a picture is very important: to protect and support an artwork and to enhance the work it surrounds. As any wooden object, the frame is vulnerable to damages: wear, biological degradation, lack of ornaments, etc. Uneven relative humidity and temperature can affect wood substrates and lead to swelling and shrinkage. The result is often a cracked and flaking gesso. Sometimes, over-gildings and over-paintings not only destroy the original details, but they make the conservation-restoration process more difficult and laborious.

A good example of case-study for restoration of gilded frame, that has a personal value for the restorer and a very interesting history, is presented in the present paper.

RESTORATION OF GILDED FRAME. A CASE-STUDY

A short presentation of the object

The object is a mirror frame, originated from a small village Pădureni, near the city Târgu-Mureş and dated about 120 years ago. The frame was a gift for the great-grandmother of the owner (Levente Majos), received from her mother-in-law at her wedding. In the 1970's the mirror was replaced with a family photo.

The rectangular gilded frame with dimensions of (600x450) mm is made of four mouldings: two horizontal and two vertical fixed together by a mitred butt joint. All four sections were assembled before the gesso and gilding were applied. On the backside there are four fillets that form a rebate to fit the mirror, the picture or the photo inside. The frame is made of spruce wood, in transition style Rococo-Neoclassic. Acanthus leaf appears to be Rococo, and the rest of the ornaments are Neoclassical. The small round ornaments that surround the inner and the outer areas of the frame are called "pearls". Surface coating seems to be gold or silver leaf with a colour transition from silver-gold in the central parts of the frame to the brown colour towards the corners.

The initial conservation state

When the frame was bringing into the restoration laboratory from Wood Engineering Faculty it presented a visible degradation. The surfaces were differently deteriorated (face in contrast with the back side, as illustrated in Fig. 1 A,B).

A B

Fig. 1.
Initial conservation state for the gilded frame (A-surface view, B- back view).

On the surface of the object the lack of gildings until the gesso layer or until the wood substrate generated lacunar areas (Fig. 2a, b). The most severe damages to the gilding finish were observed on the corners. Acanthus leaves situated in the central part of the frame were much degraded and were missing, which made difficult their reconstruction (Fig. 2b, c). The environmental conditions probably broke the decorations or completely detached them from the frame. The right upper corner was visible eroded (Fig. 2a). The joints were fragile and unstable (Fig. 2c). A more recent brown finishing layer seems to be applied on the original because the areas with lack of ornaments were covered without completion the gaps.

On the backside, a visible biological attack was observed and nails interventions (Fig. 2h). The surface was covered with an adherent dirty and smoky layer (Fig. 2f). A horizontal narrow wooden strip to fit the photo into the frame was added during a not professional intervention (see Fig. 2g). The photo was fixed on a paperboard and covered with a transparent plastic foil. The initial destination as a mirror frame was changed into a photo frame.

Some details illustrating the initial conservation state of the frame are presented in Fig. 2.

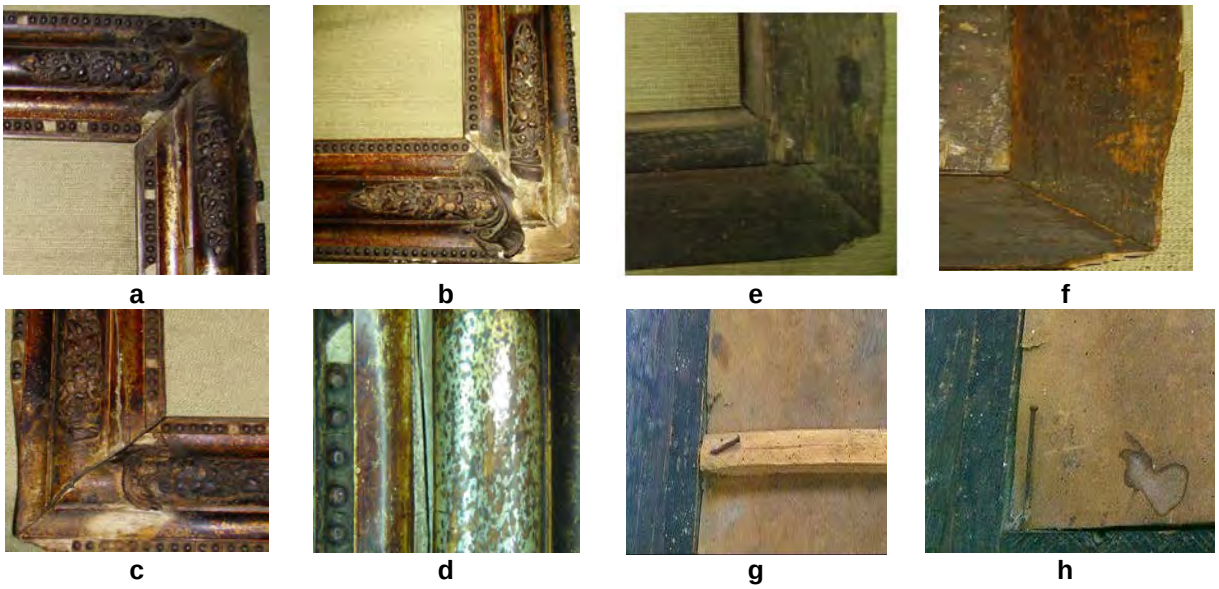

Fig. 2.
Details with initial defects of the frame (a-lack of gildings, eroded corner, b- gesso layer missing on the corner, c-fragile joint, d- finishing detail, e-visible insect attack, f- dirt and lose of material, g, h - nails interventions.

Investigation and Conservation-Restoration

Prior any interventions the frame was examined and pictures were taken to document its initial conservation state and the obvious previous interventions. The conservation - restoration concept took into consideration the preservation of authenticity. A short schedule of conservation-restoration include: consolidation of the flaking gesso and gilding, frame structural reinforcement and gluing, surface cleaning, replicating missing decorative elements, gilding, finishing.

Small gilded samples, with the flaking tendency were extracted for microscopic investigation of surface coatings. The samples were observed at different magnifications with a stereomicroscope Optika SZM fitted with a camera for image capture. The microscopic images revealed a multilayer finish consisting of white gesso, then a primer, probably bole, the silver or gold leaf layer and finally a brown film above gold leaf (Fig. 3).

Fig. 3.
Microscopic appearance of the surface -magnification 80x (a- successive finishing layers, b- cross section of finishing layers).

Before cleaning the backside wooden strip and the nails were removed and photo and paperboard were preserved. An initial, gentle, dry cleaning with soft brushes for dust removing was

done. The unsound gesso was removed and the flaking gesso was saved to be later on consolidated. Areas of detached gesso were stabilised with 3% solution of fish bone. Afterward, tests were performed to establish the best option for cleaning of the surface and the backside.

The backside was cleaned with Pronto solution for wood by brushing and wiping with soft cloth in 4-5 repeated phases until the cloth remained clean. The same cleaning solution was employed for the frame surface. A special attention was given to gildings to not flaking off.

The next step in conservation was the curative preservation against insects attack. A repeated treatment with solution of Decis was employed by injecting into the galleries. After conditioning the frail wood was consolidated with Paraloid B72 (solution of 5% in toluene) and repeated until wood saturation and strengthening.

The gluing of cracks or completion the missing wooden parts were made using bone glue 30%. Wooden strips were used to fill the large cracks (especially on the corners of the frame) after a previous cleaning of surfaces by sanding and wiping out with ethyl alcohol. Similar operation was done for gluing the backside fillets that form the rebate for mirror fitting (Fig. 4). Attention was given to an appropriate "pressing" of elements to ensure adequate contact until adhesive film formation. The excess of cured adhesive was further cleaned by moistening and scrapping away.

Fig. 4.
Sequences of the restoration before gilding (a-surfaces after cleaning, b- curative treatment and consolidation, c- gluing of wood, cracks, completion of missing parts).

After the frame was consolidated and structural reinforced, the successive gesso layers were added and the missing decorative elements were replicated according to existing models.

The gesso primer was prepared as a mixture of calcium carbonate and fish glue 6% until an appropriate viscosity ("sour cream" consistency). It fills the irregularities and other imperfections in the surface of the wood. It was applied warm to the surface to be gilded. Several coats of gesso were applied and allowed to dry before the gesso surface are worked to create detailing or to smooth the surface. After that, the surface was sanded; the dust was removed by soft brush under blowing air and fast wiping out with diluted ethyl alcohol (1:1 in water). Gesso was also the primer layer before other decorative surface treatments. Prior, a fish bone 3% was used as a primer for new gesso layer and to consolidate the old gesso.

Missing ornaments were completed in three distinct phases:

a) Replicating the "pearls" by mould technique

A former negative "pearl" mould of 10cm was made using commercial clay (Fig. 5a). After clay hardening it was sealed with shellac to create an impermeable surface. The positive gildings (Fig. 5b) were manufactured by mixing the bone glue 30%, calcium carbonate, soft paper strips (corresponding of A4 format) previously moistened and water until a plasticine consistency. It was fixed and pressed into the oiled negative form, and carefully removed after jellification of the adhesive. The positive moulding was put on the flat and rigid surface to final drying (Fig. 5c). Small, sectioned pieces containing 2-3 pearls were then gentle sanded, dusted off and glued on the frame with fish bone 10%

(Fig. 5d). Small clamps were used for fixing the pearls.

b) Carving in gesso layers the acanthus leaf according to the principle of existing model (Fig 5e) - by hand, using metallic spatula, wooden sticks or paintbrushes.

c) Re-gluing of the existing ornaments (Fig. 5f, g).

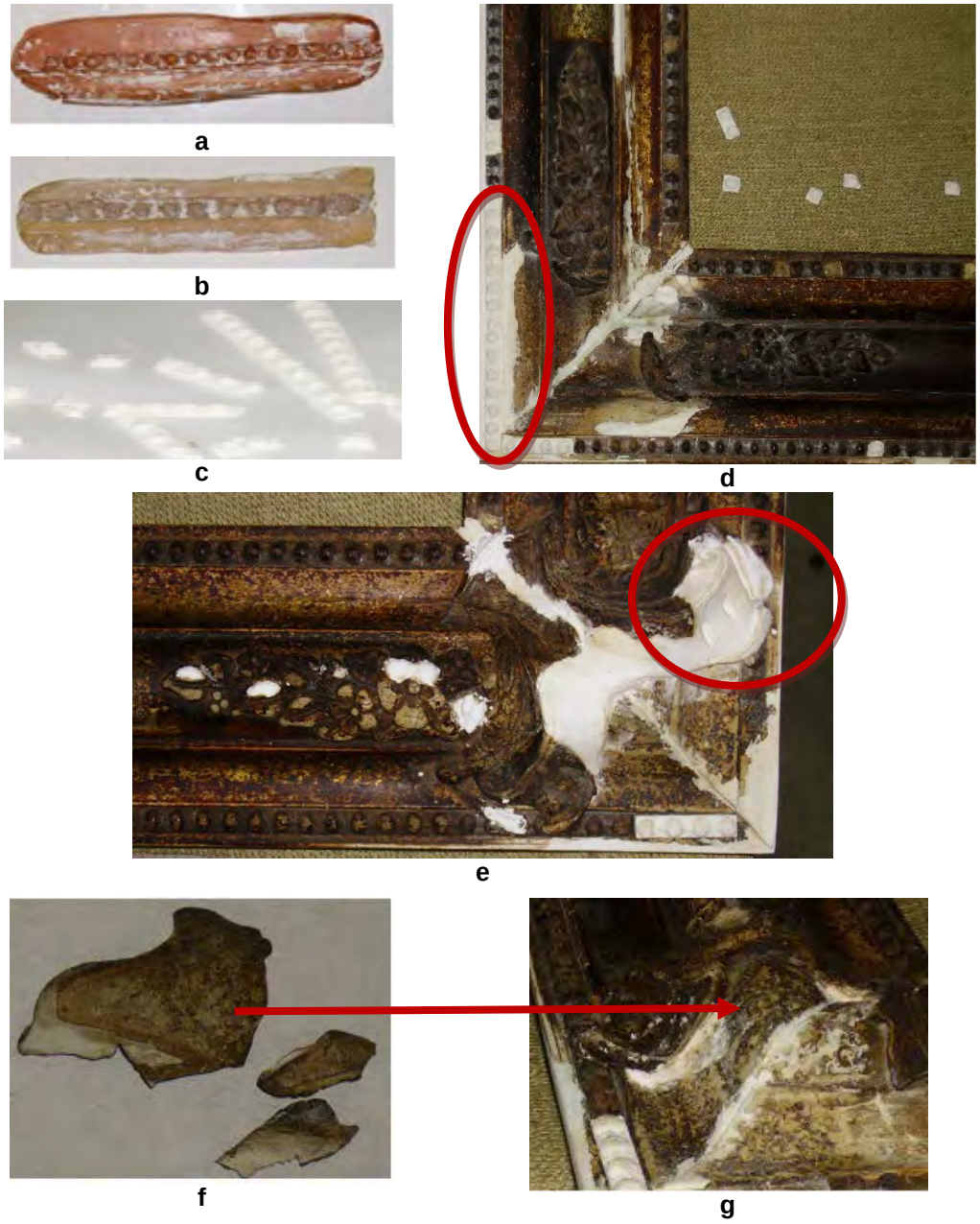

Fig. 5.
Re-gilding of missing areas
(a-negative mould, b- positive mould, c- "pearl" mouldings, d- completion of missing pearls and gesso, e-carving of acanthus leaf in gesso layer, f- flaking ornaments, g- re-gluing of the existing ornaments).

Finally, after several tests, colour reintegration was done with a brown patina in one or two layers, depending on the type of surface: one layer on the old surfaces and 2 or more layers on the new gesso and mouldings. The concave or profiled areas of the frame were then covered with a gold Liquid Leaf applied by gentle and discontinuous wiping to imitate the patina. The surface of the frame in contact with gold liquid was no more than 0.5cm^2. After 24h drying the whole object was finished with shellac (Fig. 6).

A preventive treatment with an insecticide-fungicide primer Proxilin was applied on the backside of the frame.

a

b

Fig. 6.
The gilded frame after restoration (a-chromatic integration, b-final state).

CONCLUSIONS

The conservation-restoration treatments have greatly enhanced the frame's appearance and the final result was remarkable. This case-study was a challenge for the restorer, which is also the owner of the gilded frame. Even though there was a very difficult case, it was documented and well approached, according to the basic principles of restoration.

The restored frame was displayed in different exhibitions: *Restoration 2016*- Faculty of Wood Engineering, *Researchers's Night 2016*-Transilvania University.

It must be noticed that gilded surfaces require very little maintenance other than careful dusting. For longevity of the gilded frame is compulsory to maintain stable environmental conditions. The gildings are still fragile and ongoing inspections are important. Always any surface elements that become detached must be saved.

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WOOD IDENTIFICATION OF ANTIQUE TOOLS USED FOR MANUAL PAPER PRODUCTION

Nicola MACCHIONI
Via Madonna del Piano, 10 50019 Sesto Fiorentino, Italy
E-mail: macchioni@ivalsa.cnr.it

Simona LAZZERI
Via Madonna del Piano, 10 50019 Sesto Fiorentino, Italy
E-mail: lazzeri@ivalsa.cnr.it

Lorena SOZZI
CNR – IVALSA
Via Madonna del Piano, 10 50019 Sesto Fiorentino, Italy
E- mail: sozzi@ivalsa.cnr.it

Abstract

This paper deals with the identification of the wood species used for the production of wooden tools in an ancient plant for manual paper production.

After a quick historic contextualisation of the importance of the paper industry developed in the Pescia district (Tuscany, Italy), this paper describes the process of manual paper production and the different tools, mostly wooden, used by the workers. The historic plant "Le Carte" still preserves these tools, which were sampled to scientifically identify the timber species. Samples were drawn from any wooden component of the equipment involved in the whole manual paper making process. Sampling was performed on any portion of the artefact where the species of origin was not clearly and reliably identifiable by eye, or aided by a manual magnifying lens.

In total, 1223 identifications were performed and 25 timber types observed. Most of timber identifications regard the large mould collection (434 artefacts, 1154 identifications, 17 timbers), while the equipment included fewer artefacts, identifications and timbers.

Obtained results are then technologically discussed, considering the features of each species and the specific needs of each final use.

Key words: *species identification; wood anatomy; manual paper; wooden tools*.

FOREWORDS AND AIMS

The manual paper production industry settled along the river Pescia and around the homonymous town (Northern Tuscany, Italy) from the XV century, strongly influencing both the town and surrounding countryside.

The importance of this industry is now exemplified by the establishment of "Documentation Centre of the history of paper working " which is also home of " Paper Museum Association NPO "in the building of the former primary school in town Pietrabuona. The old factory called "Le Carte" (built in the beginning of XVIII century, but active until the '90s of the XX century), purchased in June 2003 by the Association from the paper mill Magnani 2000 SpA (Bini 2012) is being converted into a museum.

The manual production of paper sheets needed several specialised workers, each characterised by a role and a skill with a specific name. Historical manual production of paper within the paper mill "Le Carte" in Pietrabuona di Pescia, used the following materials: water, as a driving force and solvent of the mixture for the production of paper sheets; macerated plant fibres and animal glue, as raw materials of the paper. The production tools were made of local stone (Pietrabuona means "good stone"), several different types of wood and cast-iron probably introduced during the nineteenth century.

Wood is involved in the tools that lead to the production of the finished sheet at every stage, with the exception of drying. The specific function of some of the tools is not completely clear, as is their assembly them, suggesting that a few decades can be enough to endanger a production process that shaped the local social community over several centuries. The re-discovery of traditional timber uses in the recent past, allows us to understand some aspects of the way of life of the past and the relation to natural environment (Melo Junior & Torres Boeger 2015).

The paper mill is nowadays under restoration, to become the site of the Museum of Paper. The building still preserves the entire original equipment for paper production, dated to XVIII - beginning of XIX century.

This equipment is: a battery of hammers, moulds, drying press and tools for sheets finishing. The watermarked mould collection is the largest one in the Museum (450 artefacts); including one mould dating to 1812 that shows the side faces of Napoleon and Marie-Louise to celebrate their wedding. Other objects, such as canvas, waxes, stamps and dies used for the production of watermarked sheets are also held within the collection. Most of the artefacts are made partially or totally of wood.

CNR-IVALSA was appointed to investigate the watermarked moulds, to identify the tree species, evaluate their state of preservation and study the methodology used to assemble the moulds. The work was preliminary to the official recording and registration within the Museum collection. Then the second step was the analysis of other wooden equipment used for manual paper production.

The general aims for both the steps were:

- understand the knowledge of wood utilisation at the time;
- know which species may be used in the course of maintenance and repair of equipment, prior to their entry in the new collection of the Museum.

The Italian standard UNI 11161:2007 establishes the proper requirements that one must take into account in the conservation, maintenance and restoration of wooden artefacts that are a part of our cultural heritage. The standard defines the essential criteria that must be followed when carrying out interventions.

Among them, the work performed for and with the Museum followed the requisites related to wood technology: wood identification and diagnosis of the state of the artefact (and its single wooden components), through identification, classification and quantification of biotic and abiotic degradations.

The aim of the paper is the description of the identification of wood species and discussion of the possible selection of species determined by their final use. Wood identification was performed following a scientifically sound path: wood anatomy is the most efficient tool to identify historically used timbers, including pieces of arts (Lisboa 1994, Kristjansdottir et al. 2001, Giachi et al. 2003, Timar et al. 2012, Ruffinatto et al. 2014, Melo Junior and Torres Boeger 2015, Macchioni et al. 2015, Mileto et al. 2016).

MATERIALS AND METHODS

The analysed wooden samples come from the watermarked moulds and the equipment for manual paper production.

The Moulds

The moulds for paper sheet production are made of a rectangular frame on which the metal cover is stretched, frequently with a woven watermark, working as a filter for the deposition of the fibre felt (Figure 2). Below the metal cover several small wooden laths, technically called ribs, act as braces to avoid the deformation of the frame (Figure 1, right); they also have a specific shape to facilitate the dripping of water.

Fig. 1.
On the left a complete mould made of the frame stretching the watermark. Above it another loose frame, called "deckle", that shapes the paper sheet. On the right the lower part of the frame showing the small wooden laths called "ribs".

Another wooden rectangular frame, loose above the mould, completes the filtering system. It is called a "*deckle*" and gives the final shape and dimensions to the paper sheets. To speed up the paper production the vatman had a couple of moulds with a single deckle (Loeber 1982) to prepare a

second sheet during the couching of the previous one, thus the collection has half the number of deckles compared to moulds.

During the diagnostic evaluation, several moulds showed traces of small joinery modifications that added new elements and different wood species to the original moulds.

The Equipment

The wooden samples were drawn from any wooden component of the equipment involved in the whole manual paper making process (Figure 2): hammers, Hollander, vat edges, pulpit of the coucher, trolleys for first drying, press, benches for the final operations and turbine of the water mill power plant.

The Sampling

The drawing of a sample must be always considered damaging for the artefact. The Italian standard UNI 11118:2004 establishes that the sampling must always be under the permission and the direct observation of the curator of the artefact.

The authors always tried to reduce as much as possible the sampling and, as stated in the standard, they were made close to damaged areas and as hidden and small as possible to reduce the aesthetic impact on the artefacts.

Fig. 2.
Three examples of the wooden equipment: a double press on the left, the battery of hammers in the centre, the pulpit of the coucher on the left.

To define the specific composition of each artefact the sampling was made on any portion of the artefact whose timber species was not clearly and reliably identifiable by eye naked, or aided by a manual magnifying lens (up to 10x). Every different component was taken into account; including the small elements added for repairs.

When the macroscopic observation was not sufficiently reliable, a portable USB microscope allowed observation of specific anatomical features on cross sections, thus avoiding further sampling.

When the macro and aided observations didn't allow a reliable level of identification, a small sample was drawn. It was then put in a marked vial that was photographed on the sampling location.

Samples were then brought to the laboratory for anatomical observation, where they were cut along three anatomical directions (cross, longitudinal radial, longitudinal tangential) and observed under an optical microscope. Cuts were made by hand using a razor on a freezing plate (Figure 3).

Fig. 3.
Hand cut of a sample by a razor. The sample is blocked on a freezing Peltier plate.

More in detail, samples are boiled until they get to the bottom. After boiling, the sample is finished preparing the surfaces corresponding to the three anatomical directions of wood, this operation is performed with the aid of a stereoscope.

The orientated sample are then put on the freezing side of Peltier plates in order to be cut for obtaining thin sections, using ice as a vice. The cut is made by hand, using industrial razor blades with rigid spine.

The thin sections obtained are finally placed on a glass slide with a few drops of glycerol and covered with a coverslip for observation under the optical microscope

To identify timbers, the observed features were compared to the descriptions in Jacquiot 1955, Schweingruber 1990, Nardi Berti & Edlmann Abbate 1992.

RESULTS AND DISCUSSION

In total, 1223 identifications were performed, and 25 timbers identified. Most of the identifications regard the large mould collection (434 artefacts, 1154 identifications, 17 timbers).

Table 1 shows the final summary of the identified timbers and their classifications according to the use within the production plant.

The following microscopic images in this paragraph were obtained during the identification of the samples. Thus, their quality is determined by the dimensions and quality of the samples and by the practical work on this kind of observation, aimed principally at identifying the timber.

Table 1

Identified timbers and their functions within the artefacts.

Timber	Artefact category	Artefact	Identifications
Silver fir (*Abies* sp. cfr. *alba*)	moulds; tools	frames; fixing components; ribs;	7
Spruce (*Picea* sp. cfr. *abies*)	moulds; equipment	fixing components; ribs; covering system of the Hollander machine	80
Boxwood (*Buxus sempervirens*)	moulds	fixing components	1
Chestnut (*Casatnea sativa*)	tools; equipment	structural components of press	4
Cypress (*Cupressus sempervirens*)	moulds; equipment	frames; fixing components; deckles; ribs; hammers components	734
Ebony (*Diospyros* sp.)	moulds	fixing components	1
Heather (*Erica* sp.)	moulds	wooden nails - pegs	3
Beech (*Fagus sylvatica*)	moulds	frames; deckles; fixing components; ribs	8
Ash (*Fraxinus* sp.)	tools	blocking system	1
Apple tree (*Malus* sp.)	moulds	deckles	1
Mahogany (*Swietenia* sp.)	moulds	frames; deckles	62
Walnut (*Juglans regia*)	moulds; equipment; benches	ribs; structural elements of press; press screws	24
Obeche (*Triplochyton scleroxylon*)	moulds	fixing components of frames	1
Elm (*Ulmus* sp.)	equipment	vat edges	1
Alder (*Alnus* sp.)	tools	shears	1
Pear tree (*Pyrus* sp.)	moulds	ribs	1
Stone pine (*Pinus pinea*)	moulds	deckles	1

Pitch pine (*Pinus* sp.)	moulds	fixing components	3
Scots pine (*Pinus sylvestris*)	moulds	frames; fixing components; ribs.	195
white pine (*Pinus* sp.)	moulds	ribs.	48
Poplar (*Populus* sp.)	moulds; equipment; benches	fixing components; press rotating wheels; benches	12
Oak (*Quercus* sp.)	equipment; tools; benches;	press structural elements;	29
Black locust (*Robinia pseudoacacia*)	equipment; tools	hammers; palette for Hollander	3
Willow (*Salix* sp.)	benches	bench drawer	1
Service tree (*Sorbus* sp.)	equipment	turbine wooden teeth	1
Total			**1223**

The raw materials selected for the manual production of paper were rags of cotton, hemp, linen and special dyes. Rags, after being cleaned, underwent a specific mechanical and chemical maceration and a reduction to the fibre grade through stacks of multiple beating hydraulic hammers.

The beating battery system that beats down the rags into pulp, was made of robust, local hardwoods: black locust and deciduous oak (*Quercus* sp., pertaining to the *Quercus* subgenus, Figure 4); some small secondary components, mud flaps, were made of cypress wood. Selected species had to be not only hard, but also heavy to apply a powerful load, beating the rags prior to maceration. Normally, in wood strength and density co-occur. Even if *Robinia* (black locust) cannot be strictly considered a member of the Tuscan flora, nevertheless is perfectly adapted to the local climate from some centuries, thus we can now call it a local hardwood (Gellini and Grossoni 1997).

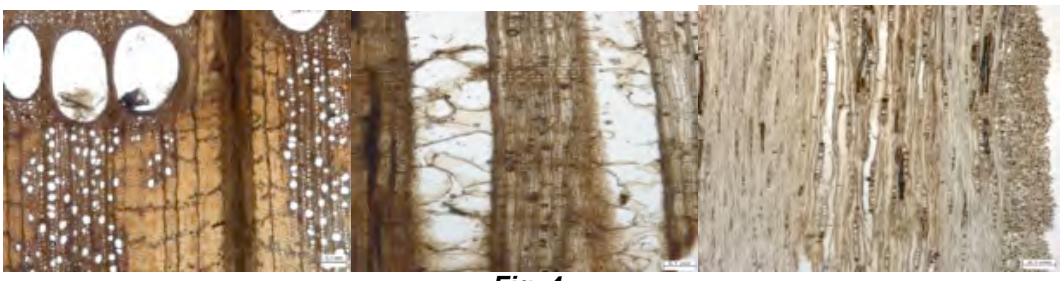

Fig. 4.
Quercus sp. a) cross section (ref. bar 0,1mm); b) radial section (ref. bar 0,1mm); c) tangential section (ref. bar 0,1mm).

Fibres were then refined by the so-called Hollander machine. The Hollander at the Museum is a stone oval basin, local made, on which the metal gears of refining system are installed. The part made of wood is the covering of the cylinder and blades of the machine. This is one of the most modern machines of the plant, thus the wooden components are just small spruce boards, likely not produced in the inner joinery of the plant. The palette used to shuffle the pulp is clearly a homemade tool made out of black locust timber.

The refined homogenised pulp, ready to become paper, was then moved to the stone vat, where it was warmed and mixed with the right amount of animal glue (gelatine). To ease the work of the vat worker, the edges of the stone vats were made out of elm and ash wood, directly fixed to the stone. A wooden pulpit, made of oak wood, further protected the work of the *coucher*.

The mixture was collected by means of special tools, the moulds already described, to produce the paper sheets. The *vatman* plunged the mould in the vat and pulled out the same amount of pulp that was then distributed over the entire surface of the metal cover: the paper sheet was obtained through a process of union between the vegetable fibres to form a uniform surface.

Felting of the fibres was obtained by means of the metal covers; they are composed of a set of small bronze wires disposed at right angles and spaced by a few millimetres between them, held in place by the chains. Vegetal fibres felt on this surface by allowing the water to drain through the wires.

The metal cover is mounted on a rectangular wooden frame (the mould). The work surface is delimited by a frame of wood, called "deckle", that is not fixed but rests on the perimeter of the metal cover to allow the sealing of the pulp and thus delimiting the size of the paper sheet that will be obtained.

Once the sheet had formed, the *vatman* passed the mould to the *coucher*. After leaving the sheet for a moment to allow the water to drain, the *coucher* reclined the mould on a wool felt, separating the sheet from the mould. This operation, reduced the water content by one half, and allowed the sheets to detach from the paper felts. According to the average productivity of a manual paper plant, the cycle of dipping and couching to produce one sheet of paper takes under 25 seconds (Loeber 1982).

Most of the work of the inner joinery was probably devoted to the production and fixing of the moulds. Some of them were clearly heavily used, because they exhibit signs of friction where they were held, marks that now seem sort of handles. Generally speaking, there are two groups of moulds: the first one is of local production, made of local species (cypress and pines), while a second, smaller group is of British production, certified by a copper brand label, made of mahogany and pine.

Cypress wood is light coloured, even the heartwood, which is resistant against insects and very durable to fungi (Giordano 1997). This feature is crucial, because the typical environment of use, very humid and warm, made the moulds prone to xylophagous but also to warping and distortion. To face this problem only clear, quarter sawn wood was used, without a single defect; moreover, the joinery had precise prescription about the treatment of cypress timber before its transformation into boards and artefacts: stems were maintained for many years, before being sawn, in the water stream of the canals giving power to the plant. Probably this long "hygro-treatment" helped in releasing the growth stresses and reducing warping when subsequently used. The corner connections of the frame elements are then tightened by vertical wooden pegs made with small branches of heather.

The British moulds were made out of a tropical timber, the mahogany (*Swietenia* sp., Figure 5) from Central America. During XIX century, British traders had a monopoly on wood trading from the Caribbean, and mahogany was the most important commercial timber from the region, specifically from Honduras (Bowett 2012).

Mahogany wood is also durable against fungi and resistant to insects (Tsoumis 1991) and the radial cut of the wood prevents warping. From the point of view of present times, it seems odd to find such a precious wood to produce an industrial tool, but at that time the availability of mahogany was still large and the price affordable. Nowadays the *Swietenia* species from Central America are protected by the Washington convention.

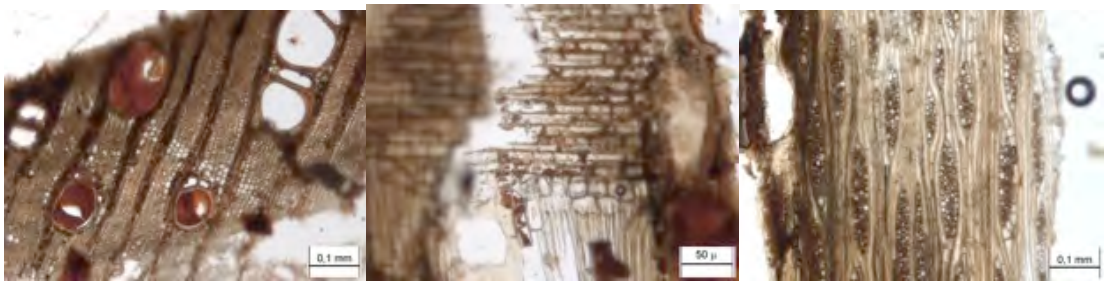

Fig. 5.
Swietenia sp. a) cross section (ref. bar 0,1mm); b) radial section (ref. bar 50 µm); c) tangential section (ref. bar 0,1mm).

Also, the small laths called "ribs" require accurate selection of the material to have elements that do not warp, ensure good mechanical performance and lightness. All the ribs are made of softwoods, the local ones were made out of local timber, like cypress, but also of timber derived from the wood trade at a national level, such as Scots/Austrian pine and spruce from the Alps. The last two woods are susceptible to insect attack and a few of those elements show severe attacks from both long horn and furniture beetles. All the British moulds possess ribs made of White pine (*Pinus* sp.), a North American softwood.

Wood species for the whole mould production must have another important technological feature: they had not to release coloured extracts when used in warm water, to not stain the fibres and the paper mat. All the listed timbers are light in colour, with the exception of mahogany, which is deep red. Nevertheless, the extractive content of the *Swietenia* species is low (around 3,5% Fengel & Wegener 1984); moreover, we could also expect a pre-treatment of wood used for mould production through a hot water washing, described by Loeber (1982).

The fibre mats drained out, shaped and dimensioned by moulds and deckles, were then laid down (couched) on damp cloths piles, as felt blankets, stacked on wooden trolleys, that were not

plane, but bent to drain down the water dropping from both paper sheets and cloths. The trolleys were made out of well selected oak wood to not rot in a wet environment. The stacks made of alternate layers of felt blankets and paper sheets were then gently pressed in manual wooden presses to increase, through pressure and drying, the transformation of the fibre mat into paper sheets.

The following phases of sheets production was then performed by three different specialised workers to separate paper from the felts: the *poster* that removed the upper felt to dispose it in the stack of felts that the *coucher* will then use in the new round of production of sheets from the vat. Then the *breeder*, who detached the sheet from the lower felt, helped by the *container*, and placed the sheet on the pile of moist sheets called *post* which was subjected to a second mechanical pressing.

The three big presses (one of them is double, Figure 2 left) that have been sampled have a constant composition, probably showing a common production. The struts are made of oak or chestnut wood, the big upper crosspiece is always made out of 2-3 elements of walnut wood (Figure 6), as are the screws and the rotating system. The lower, mobile, crosspiece, the one directly pressing on the stack, is made of oak wood. Some secondary parts of the system that allow rotation of the screw can be also made of poplar. The wooden levers used to apply the rotating movement to the screw are always made of chestnut.

Finally, the sheets were individually exposed to natural ventilation by hanging them over hemp ropes in a drying room, normally an attic with numerous large windows. This was the only production step in which no wooden tool was involved in: the drying of the sheets pulled out from the stacks was made by a simply hanging the sheets in the air, normally in the attic of the plant, the so called "drying loft".

Dried sheets have rough edges (the so-called "deckle edged sheets") that need to be cut to achieve commercial dimensions and shapes. All the final operations before selling were made with several benches and tools, frequently made out of different metals, mostly cast-iron, and wood. In most cases the timbers were poplar, willow and oak, but for some specific tools also alder, chestnut and walnut were used.

Fig. 6.
Juglans regia a) cross section (ref. bar 0,1mm); b) radial section (ref. bar 50 µm); c) tangential section (ref. bar 0,1mm).

During the XX century, a power plant was installed to produce electricity from the water that already provided power to the beating system of hammers through a camshaft. Large oak beams support the big cast-iron turbine (Figure 7). To reduce the roar of the cast-iron gears, the teeth of the horizontal turbine are made of wood and they chose service tree wood (*Sorbus* sp. probably *domestica*) due to its hardness and ability to stand the friction.

Fig. 7.
The turbine producing electricity: the teeth of the upper gear are made of service tree wood.

CONCLUSIONS

The 1223 identifications allowed 25 species or group of species to be found, among them 5 softwoods and 4 hardwoods were considered to be most abundant. 11 timbers were found only once, thus they must be considered very specific (like *Sorbus* for gear teeth) or sporadic, of almost accidental use.

The choice of the timber, apparently local, was always well related to their final use; a technological explanation for the timber choice was always clearly evident.

The most important aspect was high durability, due to the typical working environment characterised by high humidity and temperatures, making timber prone to fungi and insect attack.

Indeed, the small sapwood portions show decay caused by *Anobidae* attacks.

Local moulds were always principally made of cypress wood, with a high selection of the material, without any defects and quarter sawn to achieve dimensional stability. Imported, British moulds were always made of mahogany wood, selected with the same accuracy.

Bibliographic sources confirm the importance of the mahogany trade by the English, starting from the beginning of XIX century, principally from Honduras. Mahogany is a hardwood with a fine straight grain, that is highly humidity resistant and does not warp if well-seasoned. The British have used this material for over a hundred and fifty years, producing moulds soundly constructed and easy to handle.

All moulds should not release dying substances when plunged in warm water, to not affect the colour of the paper. Cypress does not release coloured extracts, while mahogany underwent a long treatment in hot water before being worked to become a mould.

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RESTORATION OF AN ITALIAN "*CASSONE*" FROM 17TH CENTURY

Ion Valeriu OLARU
CNM ASTRA Sibiu- Centre for Cultural Heritage
Piaţa Mică, nr. 11, 550182, Sibiu, România
E-mail: valeriuolaru.astra@yahoo.com

Maria Cristina TIMAR
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: cristinatimar@unitbv.ro

Anca Maria VARODI
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: anca.varodi@unitbv.ro

Daniela Adela VĂCARIU
CNM ASTRA Sibiu- Centre for Cultural Heritage
Piaţa Mică, nr. 11, 550182, Sibiu, România
E-mail: dana_canura@yahoo.com

Ileana CHIRTEA
CNM ASTRA Sibiu- Centre for Cultural Heritage
Piaţa Mică, nr. 11, 550182, Sibiu, România

Abstract

This paper presents the conservation - restoration process of a Cassone from the 17th century, a valuable piece of furniture, classified as thesaurus of Romanian Cultural Heritage. This is part of the George Călinescu collection, belonging currently to the Romanian Academy. George Călinescu (1899-1965) was a Romanian novelist, journalist, literature historian and critic, academician, representing an important peer of Romanian culture. Cassone defines an Italian type of highly decorated large chests, usually commissioned by rich merchants and aristocrats families from Italian culture at the occasion of their daughters' marriage. The restoration process of this object was part of an important restoration project undertaken in 2016 at the ASTRA Centre for Patrimony, the Polychrome Restoration Laboratory, from the ASTRA Museum Sibiu. The complex conservation - restoration of this valuable piece of historic furniture, which was in a precarious state of conservation, required specific scientific investigations and appropriate interventions, which are detailed in the paper.

Key words: *conservation – restoration; cultural heritage; Cassone; investigations; FTIR; microscopy.*

INTRODUCTION

Almost disappeared today from the modern life and house, dowry chests, also called marriage chests, are furniture items with a long history and international representation, with particular features for different cultures and historic periods. The surviving artefacts of this kind, coming from past generations, often inherited within families or collected, are bringing to nowadays stories and memories from another time, about other people and another world. Like a mystery box, placed in the most beautiful place in the house, the old dowry chests are revealing the social and economic status of their owners and also valuable information on the period they were manufactured, including materials and techniques alongside elements of life philosophy (Pripon 2012, Ionescu 2013).

To these outcomes contributes, with a great deal, the special and meaningful decoration, characteristic to the very different dowry chests. The craftsmen who manufactured these cherished objects were struggling to carve or paint these chests in a very special way, combining aesthetical features with symbols. Wooden chests, from all over the world, have been carved, painted or inlaid with colourful woods and other materials (e.g. mother-of-pearl) for centuries (Stone 2015).

A very special type of dowry chests is represented by the *Cassoni.* Cassone is the specific name given to an Italian type of highly decorated dowry chests, usually commissioned by rich merchants and aristocrats' families at the occasion of their daughters' marriage. The name originates

from a small locality in northern Italy. Cassoni were employed between 14th and 18th centuries as ceremonial and representational objects in the wedding procession (Schubring 1915). The cassone ("large chest") was the most important piece of furniture of Italian Renaissance, which represented the bride's parents' contribution to the wedding, being at that times one of the trophy furnishings of rich merchants and aristocrats in Italian culture (Cionca 2004 a,b; Ajmar-Wollheim and *Dennis 2006*).

The lids were usually decorated with the names and/or the coats of arms of the noble families who were marrying their children. Such artefacts were also given, in the rich families, to the daughters at the occasion of their religious confirmation. Cassoni were richly decorated, usually by modelling and carving in gesso, gilding, painting, carving in wood and polishing with resins. Rich Italian families hired the great artists of the time to decorate these pieces of furniture. Among those artists there were: Apolonia di Giovanni, Paolo Ucello, Donatello, Andrea Mantegna, Filippino Lippi, Francesco di Giorgio Martini, Beccafumi (Robbins 2004, www.britannica.com/topic/cassone, www.brown.edu/Departments/Italian_Studies/dweb/arts/cassoni). These characteristics make cassoni valuable artefacts with artistic and historic importance, part of world cultural heritage, being, therefore, highly appreciated collectible items for both specialised institutions and private owners.

In 2016, the ASTRA Centre for Patrimony, from the ASTRA Museum in Sibiu, restored fourteen pieces of valuable historic furniture, from the George Călinescu collection, currently belonging to the Romanian Academy. Among those pieces there was a cassone, dated from 17th century, listed as treasure in the official list of Romanian National Cultural Heritage.

George Călinescu (1899-1965) was an important Romanian novelist, literature historian and critic, representing a well-recognised peer of Romanian culture, who became member of Romanian Academy in 1949. Educated and refined intellectual, G. Călinescu appreciated and collected during his life various art objects. His literary work actually reveals his taste for art objects by the frequent and detailed description of the interiors, often inspired from autobiographical events. An eloquent example is the novel "Black Chest", inspired by a piece of furniture bought after a war from a flea-market, in which he discovered the archive of an ancient family.

The fourteen restored furniture pieces will recreate at the G. Călinescu Memorial House Museum in Bucharest the inspirational atmosphere from the writer's office. Every piece of furniture has an artistic, historical, technical and sentimental value, eight of which being classified as National Cultural Heritage Treasures. One of these eight pieces is the "*Cassone*" presented as case study in this paper.

OBJECTIVE

The main objective of the research work presented in this paper was the restoration and conservation of a valuable *Cassone* dowry chest, dated from 17th century, in accordance to the conservation-restoration principles and code of practice. For this purpose adequate documentation and employment of diverse analytical techniques were necessary to understand the object, evaluate the conservation state and select the appropriate intervention methods. This work is part of an important project undertaken in the year 2016 by the ASTRA Centre of Cultural Heritage from Sibiu (Romania), within the Laboratory of polychrome wood restoration

PRESENTATION OF THE OBJECT

The *Cassone* (Fig. 1 – initial state – before restoration) presented as case study in this paper belongs to the Romanian Academy, being part of the patrimony of the George Călinescu Memorial House Museum in Bucharest. This object is included in the official list of cultural heritage artefacts (by CIMEC – code 2739/18.11.2004) in the thesaurus category, as a guild coffin in German Gothic style, made of oak wood, dating from 17th century, though it seems to us to meet rather the characteristics of a dowry chest of cassone type, specific to the occidental area (Italy) during that period.

The object is a large wooden chest with rectangular shape (1630 × 630 × 66a mm), supported by short legs, the two from the frond being shaped as lion's paws. The plan lid is made from two timber pieces glued together and its width is exceeding the width of the lateral sides, coming a little bit to the front. It seems that the object also served as a bench for sitting (from CIMEC records).

The front of the coffin is decorated by sculpture in wood, with a vegetal motif representing the "*tree of life*", specific to that period of time. This is largely developed horizontally on this artefact, covering the whole front panel of the coffin as two symmetrical acanthus leaves coming out from a vessel with two ring-shaped handles. The "*tree of life"* motive, with very different representations, is one of the most important and frequently employed symbols that enrich dowry chests / furniture pieces, by painting, engraving or carving. This symbolizes the life in continuous evolution, from birth to death and regeneration, the universe, the micro- and the macrocosm, as well as immortality. It also represents a connection between earth and sky (Eliade 1994, Olaru 2014). This type of symbolic

ornament may include lateral, symmetrically distributed, other ornamental elements, such as flowers and birds.

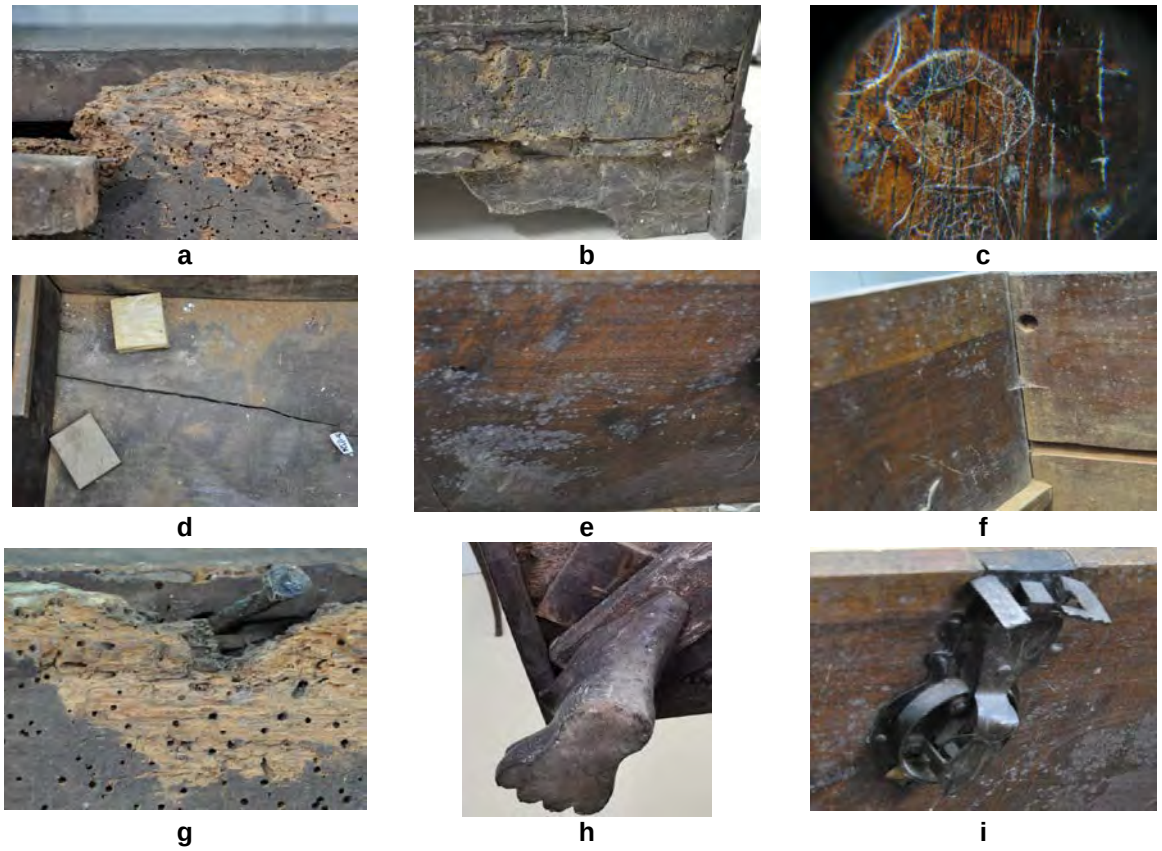
a b

Fig. 1.
Initial state of "Cassone" dowry chest: a) front view; b) the back of the chest.

A simple metal lock, ornamental lateral handles and two hinges were the metal accessories.

Initial state of conservation

The initial conservation state of the dowry, illustrated in Fig. 2, was unstable and relatively precarious due to the active insects attack, mostly in the bottom parts of the coffin and the back panel (Fig. 2a, b, g). The presence of sawdust in the numerous flying galleries (50 -120/100cm^2), of diameter of 2-3mm, indicated an active attack, most likely by *Xestobium rufovillosum*. The combined biological attack by insects (predominant – Fig. 2a, b, g) and fungi (Fig. 2e) caused wood frailness, ruptures, cracks, loss of material (Fig. 2b, g). The whole artefact was very dirty, with lot of dust deposits; clogged dirt and different spots on both external and inner surfaces. The surface of the object presented functional wear by faulty handling, cracks (Fig. 2d) and advanced structural degradation of wood elements (Fig. 2b).



a b c

d e f

g h i

Fig. 2.
Initial state of conservation: a) insects attack; b) degraded wood and erosion of bottom parts of legs with advanced surface texturing; c) aspects of aged finishing protective layer (image by magnifying glass); d) aspect of cracks; e) fungal attack, f evidence of missing small inner wooden box, dirt, spots; g, h) inadequate previous interventions; i) rusted metal locker, dirt.

It was also observed that a constitutive part was missing from inside (Fig. 2f), very likely the small inner box, characteristic to most of the dowry chests. The finishing protective layer was rough, quite thick, aged, crackled, with mirror like areas due to inadequate wetting of the support (at application phase and/or due to ageing) and low / non uniform adherence (Fig. 2c). All these strongly suggest a refinishing of the artefact.

Wood and structural degradation caused previous, totally inadequate rough "repairs", with negative impact. Previous interventions consisted in structural consolidation with metal nails (Fig. 2g) and reconfiguration of legs assembly (Fig. 2h). Metal elements (metal lock, handles) were rusted and clogged with dirt, losing their functionality (Fig. 2i).

INVESTIGATIONS AND CONSERVATION - RESTORATION

Before any direct intervention relevant photos were taken to document the initial state of conservation and the whole object was meticulously examined. The previous interventions were documented by photos and described, as these were not mentioned in the available conservation records. In the process of conservation - restoration the basic principles of good practice were respected. Authenticity of the object was preserved, interventions were based on scientific investigations, being accomplished with traditional or compatible materials, which ensure also their reversibility.

At the same time, inappropriate interventions, such as consolidation with metallic nails, were amended. So it was decided to dismantle these elements. In the same context, the inadequate reconfiguration of legs assembly and strong fragility of timber elements from the bottom of the *Cassone* justified the decision of dismantling this part for a correct approach in the restoration process and for a better cleaning.

Scientific investigation of wooden species

As first step in the process of restoration, dusting of *Cassone* dowry was made with soft brushes. Thorough examination of cleaned wood surfaces revealed that the chest was actually made two from different wood species. To establish the wooden species, small samples of wood were taken for both type of wood identified by macroscopic view (Fig. 3a, b).

a **b** **c**

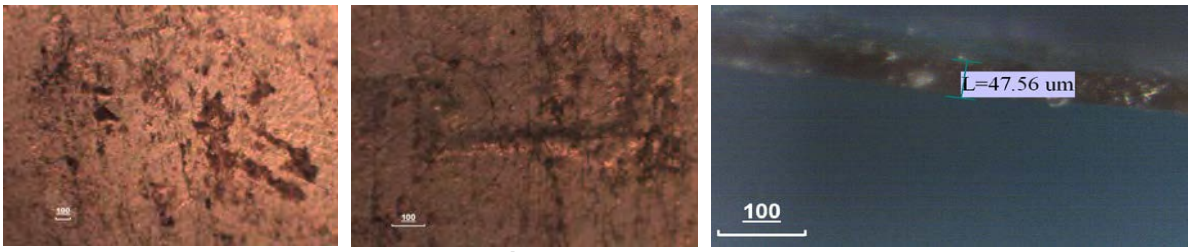
Fig. 3.
Samples extraction: a) wooden sample from the back of the chest; b) wooden sample from the front of the chest; c) coating film sample extracted for investigations (image by magnifying glass).

The wooden samples were prepared as thin transparent microscopic slides and investigated by optical microscopy (at ASTRA Sibiu). This revealed that the back and the bottom of the dowry were made from oak wood (*Quercus cerris*), while the front, the laterals and the lid of the chest were made from walnut wood (*Juglans regia*). The registered micrographs were analysed according to the microscopic identification keys and compared with reference samples from an electronic catalogue (Timar 2008).

Scientific investigation of finishing layer

The protective finishing layer was also investigated. Small film samples were taken (Fig. 3c) in order to be investigated by: optical microscopy, solubility tests and FTIR spectroscopy.

The microscopic images were recorded with Optika SZM type Olympus SZ-CTV microscope provided with imaging software. In Fig. 4 are presented the captured images for the film samples at different magnifications (40X and 90X, general view) and cross section (90X). It can be observed that the protective film, of about 50µm thick, has a pretty homogenous microstructure, with some dark brown inclusions, possibly brown pigments.



a b c

Fig. 4.
Microscopic images of film layer sample: a, b) general view (a- 40X magnification; b- 90X magnification); c) cross section (90X magnification).

To establish the nature of protective layer some dissolving tests with ethylic alcohol were made under microscope (Fig. 5).

a b c

Fig. 5.
Microscopic images registered during solubility test in ethylic alcohol (98%) under the microscope: a) 40X magnification; b, c) 90X magnification; red marks highlight some dissolution of a component with film forming properties.

Analysing the images it can be stated that the sample is not soluble in alcohol, but there is a component slightly soluble in concentrated ethylic alcohol, which migrates from the coating film. This seems to be, apparently, concentrated on the back of the film (the area of low adherence to the original finish). It can be noticed that this soluble component migrates from the sample (Fig. 5c) to the margins of glass lamella (area marked with red in Fig. 5a and 5b), forming a very thin film.

In the same context FTIR spectrometry analysis was performed employing a Bruker Alpha spectrometer equipped with the ATR unit. The spectra resulting from 24 scans were recorded in the range 4000-400cm^{-1} at a resolution of 4cm^{-1}. The processing of registered spectra was carried out with the dedicated OPUS 7.2 software. The spectra shown in figure 6 are the average of three recorded spectra (replicates). This was compared with reference spectra of traditional coating materials, such as shellac, beeswax, linseed oil from the ICDT (Brașov) laboratory collection.

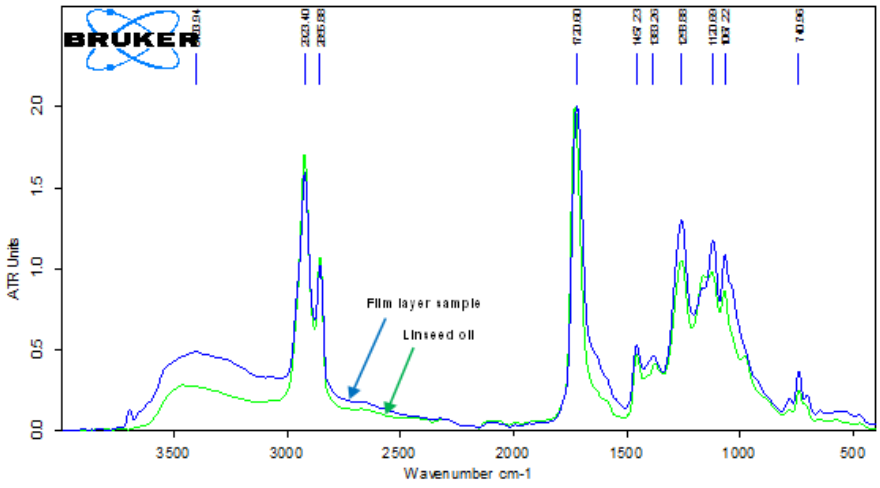


Fig. 6.
FTIR spectra registered for the protective film layer (blue), compared with linseed oil reference spectra (green).

FTIR investigation clearly confirmed that the finishing layer cannot be shellac or another related resin, as a powerful characteristic absorption at around 1153cm^{-1} is missing. On the other hand, the recorded spectra match perfectly with the reference spectra of linseed oil film, suggesting a siccative oil as the main component of the finishing layer. These observations are supported by the dissolving test in ethyl alcohol, as shellac would have been soluble, whilst cured drying oil films are perfectly resistant to alcohols and generally to solvents, making them very difficult to remove from a finished surface.

Moreover, FTIR investigation of the film formed on the glass lamella following dissolution in ethyl alcohol of the soluble component from the coating film, indicated that is most likely beeswax (Fig. 7).

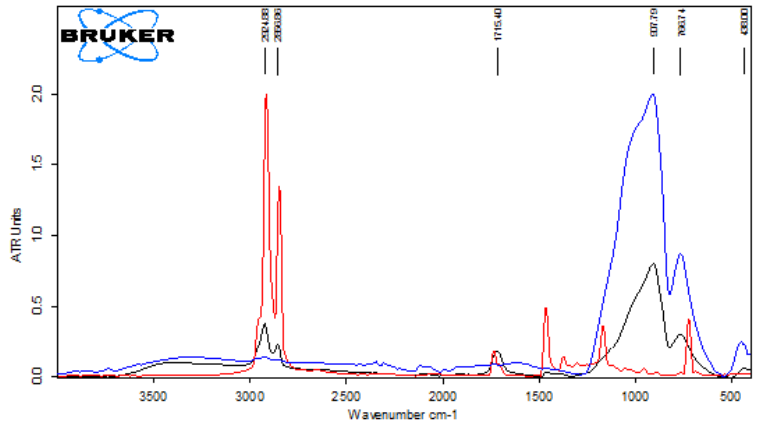


Fig. 7.

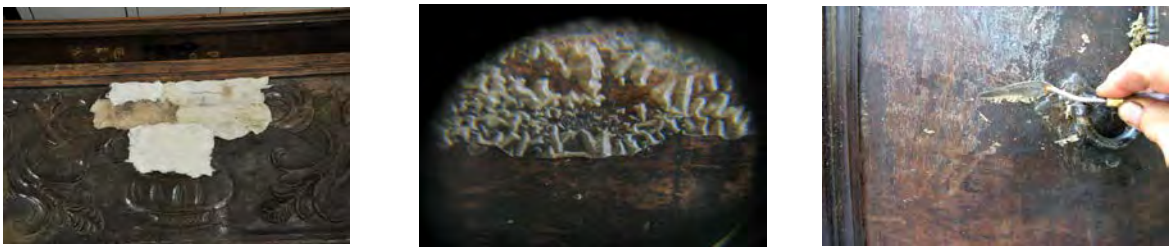
FTIR spectra registered for the film (on glass lamella) formed by the component dissolved in ethyl alcohol (black), compared with beeswax reference (red) and glass lamella (blue).

From the corroboration of all these results resulted by: microscopic analysis, solubility tests in ethanol and FTIR, it appears that the non-original finishing layer is very likely a siccative oil that has been applied to a waxed surface. This would also explain the inappropriate adherence, revealed as areas with mirror aspect due to the tendency to exfoliate from the substrate, due to the lack of adherence.

Conservation - restoration process

Tests with different solutions were made in order to establish the most appropriate solutions for cleaning and removing the non-original, aged finishing layer, which were performed directly on the object. These showed that the finishing layer was insoluble in turpentine and had a low solubility in 99.8% methyl alcohol, 93% ethyl alcohol and iso-propyl alcohol. The film was not dissolved in the tested alcohols, but rather softened by their absorption. These tests confirm the results presented above. The areas were solubilisation tests were performed as well their results were recorded and documented by digital photography.

Following these tests, it was decided to remove the thick finishing layer with different types of scalpel, blades, brushes and chemical cleaning. First step consisted in application of compresses with iso-propyl alcohol for 10-15 minutes (Fig. 8a) and after that period the soaked film (Fig. 8b) was removed mechanically with different blades (Fig. 8c).



a **b** **c**

Fig. 8.

Steps undertaken to remove the non-original finishing layer: a) application of compresses with iso-propyl alcohol; b) aspect of the soaked film (image by magnifying glass); c) mechanical removing of the soaked film.

An important step in the process of conservation - restoration was curative protection that was achieved by repeated injections with Per-xil 10 insecticide into the flight galleries (repeated injections at an interval of at least 24h). Strengthening of fragile wood areas was achieved by repeatedly injecting Paraloid B72 solution in ethyl acetate, employing successively solutions with progressive concentration (3%, 5%, 7%).

Detached wood areas were glued with rabbit skin glue (30% solution) and consolidated with wood tenons. The assemblies were kept under pressure for 24 hours. The gaps in the wood structure were filled with remedial elastic putty based on rabbit glue, oak sawdust, mountain chalk and pigments. Acrylic stucco of brown brick colour was also used. The flatness of the remediated areas was restored after drying with fine abrasive paper or cork stopper.

Also, it was necessary to manufacture a new wood small box to complete the missing one. The legs have been assembled with the chest by completing missing elements with new wood material. Chromatic integration was performed differently depending on the area, with water-based colours by fine lines and points. The chest was finally finished with beeswax in withe spirit solution, with a concentration of 20%, applied by brushing and then polished (after drying) with fine cotton fabric.

The metal elements (hinges, lock, key) have been mechanically cleaned with appropriate instruments (scalpel, steel wool). Fertan was used to protect them. The final state of the restored cassone can be seen in Fig. 9.

Fig. 9.
Final state of the restored Cassone.

CONCLUSIONS

Following a complex restoration and conservation process the valuable Cassone from the G. Călinescu collection, currently belonging to the Romanian Academy, has regained its integrity and original beauty, while maintaining its authenticity, patina and history.

The official classification of this artefact as treasure of cultural heritage was the outcome of a specialised expertise highlighting its artistic, historical, technical, documentary and sentimental significance. The former owner, George Călinescu, is an important personality of Romanian culture. Moreover, the patrimonial value of this furniture piece results from its very nature of a special type of dowry chest, an Italian Cassone, high value collectible items.

The complexity of the entire conservation-restoration process in terms of materials and technology of interventions was determined by the object itself and the advanced and the diverse degradation phenomena and deterioration. The precarious initial state of conservation was related to the variation of environmental conditions over the time, inappropriate maintenance, the inadequate repair interventions and the functional (miss)-use of this valuable artefact.

The restoration of such objects, containing old materials and technologies, involves thorough documentation /research on the specific materials, technologies, historical period and cultural production areas. This knowledge, complemented by laboratory investigations, is absolutely necessary for a scientific restoration project, specific for each object.

FTIR spectroscopy combined with microscopic technique has provided the basic information on the morphology and possible composition of the non-original finishing layer, supporting and assisting effective restoration interventions. Comparison with reference data allowed materials identification, highlighting the importance of such data-bases in the conservation-restoration practice. However, multiple investigation techniques and corroboration of results is required for reliable conclusions.

Last but not least, cooperation between specialists, laboratories and institutions serve the ultimate desire and outcome: conservation of cultural heritage.

ACKNOWLEDGEMENT

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OLD WOOD RECOVERED FROM CONSTRUCTIONS – FROM SCIENTIFIC CHALLENGE TO DESIGN OPPORTUNITIES

Maria Cristina TIMAR
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581,
E-mail: cristinatimar@unitbv.ro

Emanuela BELDEAN
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: ebeldean@unitbv.ro

Anca Maria VARODI
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: anca.varodi@unitbv.ro

Ioan MUSCU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: ioan.muscu@unitbv.ro

Abstract

A fragment from an old beam from a Saxon house in Ghimbav – Brașov, bearing the inscription 1733 J, offered the opportunities to study/ investigate degradation and ageing phenomena on old wood elements recovered from constructions by colour measurements and FTIR. The results demonstrated different ageing phenomena from the surface towards the inner parts of the wood. A core of basic principles to be considered in modern design approaches of reinterpretation and reintegration of such elements, based on three fundamental phases has been defined. Adequate active conservation methods allowed stabilisation of the material, preservation of historic information and old patina, while highlighting the special aesthetical features of such elements.

Key words: *old wood; degradation; colour; FTIR; recovering; reinterpretation; reintegration; design.*

INTRODUCTION

Wood has been from ancient history to modern times, the most important material in humans' life, with various applications from constructions to furniture and from tools and artisanal pieces of equipment for traditional household occupations to artefacts and/ or decorative elements with spiritual significance and artistic value. That is why old wood constructions and artefacts represent worldwide an important part of cultural heritage, which should be acknowledged, understood and conserved in accordance to the technical principles and code of ethics. Preserving authenticity and patina, ensuring reversibility of interventions and possibility of further treatments, alongside thorough documentation and a scientific approach, are well recognized demands of conservation.

However, the interest in old wood should not be limited to the constructions or artefacts recognized as cultural heritage. Wood, which has been in use for long periods of time, in specific conditions depending on utilization, is bearing valuable scientific information on the in-time behaviour of wood of different species in direct relation to the real in-use conditions, including incidence of degradation phenomena and effects of natural ageing (Matsuo *et al* 2010, Kacik *et al* 2014, Kranitz *et al* 2016). Old wood represents in this context an important reference for research, especially durability studies and laboratory accelerated ageing tests. Life-time prediction of wood in certain uses, as well as design of ageing tests and validation of resulting data, as their interpretation, including calculation of acceleration indexes, rely on this kind of references / comparative data (Matsuo et al 2010).

Moreover, recovered old wood is bearing a certain economic value. Several alternatives of re-using or recycling, depending on its conservation state and physical and mechanical properties,

affected both by degradation and ageing, may be considered for both economic and ecological reasons (e.g. Kranitz et al 2016).

Recovered old wood elements, after thorough and adequate active conservation treatments, may be reintegrated in new products or decorative ambient items, with exquisite and unique aspect. This approach often highlights the "beauty of degradation and wearing" in an original way, but a very fine line between real artistic value and non-value (kitsch) should be considered. Co-operation of conservators, scientists and designers is a key factor for a successful approach.

In some cases an old wood artefact or dispatched old wood elements recovered from constructions/ demolishing can cumulate historic information and/ or artistic value with a great potential for gathering important scientific data on degradation or ageing of wood. Then a multiple challenge stands in front of scientists, conservators and designers as a team to make the most of it.

OBJECTIVE

The main aim of the research presented in this paper was to explore the opportunities to study/ investigate degradation and ageing phenomena on old wood elements recovered from constructions. Moreover, it was intended to define a core of basic principles to be considered in modern design approaches of reinterpretation and reintegration of such elements. The case of a fragment from an old beam from a Saxon house in Ghimbav –Brasov, bearing the inscription *1733 J*, has been considered for exemplification.

EXPERIMENTAL RESEARCH METHODOLOGY

The methological concept of the experimental research was conceived in order to balance the scientific interest with the conservation goal. This was inspired by the case study itself, which cumulates historic and technical information with aesthetical features on a piece of old and dated wood artefact, offering a rare chance to perform scientific investigations on ageing and degradation.

Presentation of the case study

A general view of the old beam fragment considered in this research is presented in Fig.1. This was recovered from a Saxon house currently under rehabilitation work. Though the surface was very dusty and covered in dirt and debris, the existence of an inscription, denoting its historic value, was visible, as well as several defects, such as: forms of biological attack on the surface and a certain depth, areas of frail wood and deep cracks, alongside lots of rusted nails inserted during the long life-time in use. The inscription became far more clearly visible after a thorough cleaning. Microscopic investigations (not presented in this paper) demonstrated that the beam was made from fir wood (*Abies alba* Mill).

Fig. 1.
General aspect and details of inscription and degradation and of the old beam (1733) fragment.

Sectioning and samples for investigations

In order to address the research interest while also conserving the historic information, the beam was sectioned as illustrated in Fig.2. Five slices (G1.A-G1.D), with thickness of 25-50 mm, were cross-cut from one of the ends. They offered the chance to investigate and compare ageing and degradation on the surface of the beam and also on the whole cross-cut, at different depths from the surface. Moreover, four longitudinal slices (G1.0, G1.1, G1.2, G1.3), each of them 25 mm thick, were cut longitudinally from the face opposite to the one bearing the inscription. These were used to determine the variation of the density and mechanical properties from the surface layer to the inner layers of the beam in correlation to degradation and ageing (to be published elsewhere). The whole concept of beam sectioning was meant to serve the aim of this research by providing adequate samples for different investigations in a way to obtain relevant results that could be correlated.

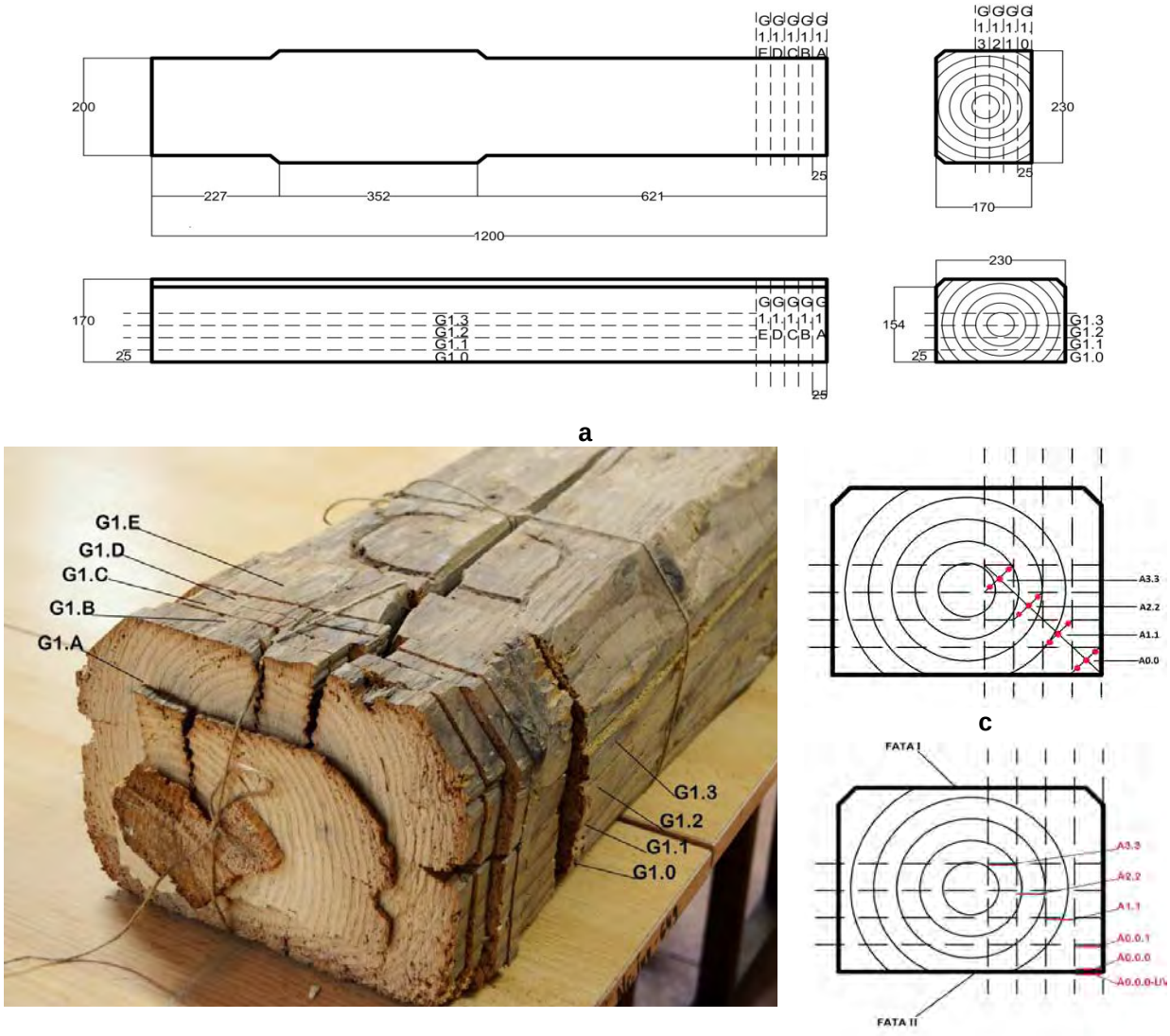

Fig. 2.
Sectioning of the beam (a,b) and drawings showing the zones of colour (c) and FTIR (d) investigations in order to highlight external and internal degradation and ageing.

Investigation methods and equipment

Colour measurements were performed in the CIELab system employing an AVA Spec USB2 spectrometer equipped with an integrating AVA sphere. Colour was measured on the four external faces of the beam (on the G1.E slice) and on the cross-sections (G1.A-G1.D) in four areas (Fig.2.c) from the outer layer (code 0, thickness 25 mm, corresponding to G1.0) towards the centre of the beam (layers 1,2,3 - corresponding to G.1-G.3) up to a depth of 100mm from the surface. On each measuring area and each slide/surface, colour was measured on minimum 3 points, actually small

circular areas of 8 mm diameter. The average values of the colour coordinates: L*- lightness (varying from 0 for black to 100 for white), a* (red-green coordinate) and b* (yellow-blue coordinate) were then calculated for each type of investigated area.

Colour differences ΔE* (according to equation 1) were then calculated to better highlight colour changes associated to ageing. The average value of 120 measurements on new fir wood samples (30 samples, each measured in four points) was employed as reference value.

$$\Delta E^* = \sqrt{\left((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right)} \quad (1)$$

FTIR investigations were performed employing an ALPHA Bruker FTIR spectrometer equipped with ATR unit. The aim was to highlight the specific chemical changes associated to wood ageing, as a function of the active ageing factors, revealing also how deep inside the wood can be registered those changes. Therefore, small samples were extracted from the areas coded A0.00-UV, A0. 00-0 (outer skin layer of the beam of about 1 mm, face directly exposed to UV and reverse of the sample) A0.01 (25 mm from the surface), A1.1, A2.2, A3.3 (areas situated at 50, 75, 100 mm from the surface on the cross-section of the beam), as presented in Fig.2d for the slice G1A.

Spectra were registered in the range 4000-600 cm^{-1}, at a resolution of 4 cm^{-1}, each spectrum resulting from 24 scans. Three FTIR spectra were registered for each type of sample/area and these were further processed employing the OPUS dedicated software (smoothing, normalisation, computing average spectra). Interpretation was based on the assignment of absorption bands according to literature and specific calculations (integrations, ratios of specific absorption bands).

PRINCIPLE ASPECTS OF REINTERPRETATION AND REINTEGRATION

Attention towards wooden elements recovered from diverse real applications (at the phase of their deactivation) and their consideration as an alternative source of wood material implies an opportunity of activating capabilities of imagining new situations, combining / reorganizing the existing material in new schemes and concepts to generate visual perceptions, emotions and inedited ambient manifestations,

Fig. 3.
Schematic principle of valorising recovered old wood by creative conceptual reconfiguration (reinterpretation, reintegration).

The configuration of the material identified, selected and considered as a creative stimulus is heterogeneous and often very particular. These configurations could be grouped as follows:

- Parts and elements from diverse wood structural configurations;

- Parts and elements from different wooden closing and cladding structures;
- Parts, decorative elements or wood ornaments in linear and flat configurations or diverse volumetric shapes, as individual or glued/assembled elements;
- Semi-finite wooden products, standardised or not, in different configurations and conservation states;
- Wood finite products;
- Wooden products and objects out of order or classified as structurally and functionally degraded;
- Fragments or parts originated from all the previous categories.

At a general overview, the creative process which includes reintegration and reinterpretation of recovered wood elements could be understood as concatenated actions, structured in three fundamental phases: the existing configuration, transformation and the new configuration (as presented in Fig. 3).

THE EXISTENT CONFIGURATION – involves a first evaluation and identification of their qualities, characteristics and particular aspects as they are given and highlighted. The information could be registered on the following aspects:

Dimensional characteristics/ proportions;
Shape characteristics;
Structural characteristics/ 3D configuration;
Textural and chromatic values;
Transformations and morphological modifications.

These aspects identified by direct visual examination or scientific investigations, as exemplified in this paper, are analysed and evaluated in order to identify the actual causes of the observed phenomena. Conclusions are organised and rated as result of the following phenomena:

Ageing;
Degradation by biological or other factors;
Accidental modifications.

Therefore, an important issue is to understand these phenomena and how they will evolve in time affecting the registered qualities and characteristics of the material, respectively to identify stabilisation possibilities.

THE TRANSFORMATION- involves a complex process of creation and imagination that cannot be standardised. This is a particularly dynamic process, influenced by the inspiration of the research team, creative abilities of the team members, work methodology and implementation instrumentation, the aim and the approached subject, requirements and instructions from third parties.

THE NEW CONFIGURATION- represents the variety of the solutions conceived and applied. The result subscribes within three fundamental directions:

- Reuse;
- Reintegration;
- Reinterpretation.

The final ideas of these configurations could be understood as "*Augmented positive compromises*" resulting from different possible approaches:

- Utilisation of the element without any intervention;
- Modification of the element by interventions and processing: mechanical, chemical, artistic, or other type;
- Combination of the element with other materials by industrial or handicraft processing, marquetry, upholstery, collage.
- Fragmentation of the element and its utilisation as constituent in composite or mixed materials/ structures.

All these approaches have as main purpose re-valorisation of the recovered wooden elements as a whole or of some of their characteristics, defined by functional- practical or suggestive-plastic aspects, which can define new interpretations and new stylistic orientations.

RESULTS AND DISCUSSION

Ageing *vs* degradation revealed by scientific methods

Colour measurements

Ageing of wood is associated with more or less advanced colour changes, depending on the wood species, the ageing conditions/ factors and the ageing time. Actually, colour measurements in

the CIELab system and calculated colour differences (ΔE) following ageing have been considered as the most sensitive ageing indicators (e.g. Timar et al. 2016 and literature cited herein), being extensively employed worldwide for monitoring ageing phenomena.

The colour differences between new fir wood and old fir wood was obvious at direct examination, being also well reflected in the data from Table 1. This data also reveal the colour differences between the outer surface, directly exposed to light, and the inner layers at various depth from the surface. The colour difference values ΔE, calculated reported to new fir wood, were 35.8 units for the surface and significantly lower for the inner layers, decreasing from 20.3 units in the sub—surface layer to about 11.0 units at a depth of 100 mm from the surface. This clearly show that ageing effects are decreasing from the surface inwards the wood, but they are still present in the inner part of the wood beam.

Table 1

Colour coordinates in the CIELab system for new (control) and aged fir wood (Abies alba* Mill.*) - measurements on an old beam (d.1733) surface and inner layers

Colour coordinates	**Control Fir** (new)	**Aged - surface**	**Aged - Inner layers**			
		A0.0-UV	**A0.0**	**A1.1**	**A2.2**	**A.3.3**
Depth, mm	-	*0*	*1 to 25*	*26 to 50*	*51 to 75*	*76 to 100*
L* (lightness)	**81.80**	**46.10**	**62.29**	**68.58**	**70.02**	**72.41**
st. dev.	*1.00*	*1.44*	*0.56*	*0.54*	*0.52*	*0.83*
a* (redness)	**4.65**	**6.50**	**6.52**	**6.62**	**6.50**	**6.20**
st. dev.	*0.43*	*0.85*	*0.59*	*0.41*	*0.38*	*0.34*
b* (yellowness)	**17.81**	**15.47**	**23.07**	**23.57**	**23.29**	**23.32**
st. dev.	*0.86*	*2.09*	*0.62*	*0.71*	*0.66*	*0.62*
Colour difference	-	**35.82**	**20.29**	**14.55**	**13.12**	**10.99**

FTIR investigation

The FTIR spectra in Fig.4 highlight the specific absorption bands of wood components in the fingerprint region (1800-800 cm^{-1}) and the chemical modifications brought about by ageing, on the surface of the beam and inside the beam as a function of the depth from the external surface. In the spectrum of the control (new, sound fir wood) could be easily differentiated the absorptions related to carbohydrates (hollo-cellulose) at 1370 cm^{-1} (CH deformation), 1150 cm^{-1} (C-O-C bridge vibration), 1024 cm^{-1} (CO stretch of pyranose rings – maximum absorption band employed for all spectra normalisation), alongside the absorption at 1730 cm^{-1} (unconjugated carbonyls) related to the hemicelluloses (actually the acetyl groups in their structure) and the small shoulder at around 900 cm^{-1} assigned specifically to cellulose. Lignin related main absorption bands are those at around 1506 cm^{-1} and 1600 cm^{-1} (aromatic skeletal vibration of lignin) and the distinctive band at 1262 cm^{-1} (guaiacyl ring typical for softwoods lignin).

When looking to the other spectra in Fig.4, all referring to aged wood, a clear differentiation is obvious between the spectrum registered on the external face (A.00-0-UV directly exposed to light) and the spectra registered for the other samples representing inner layers from the immediate proximity of the surface (A.00-0), inwards the wood material, up to a depth of about 100 mm from the surface. The FTIR spectrum of the external surface (A.00-0-UV) show, compared to both the control and the other aged samples, a drastic reduction of lignin content (decrease of absorptions at 1506 and 1262 cm^{-1}) due to UV degradation. The decrease of the bands at 1370, 1150 and 1730 cm^{-1}, whilst the band at 900 cm^{-1} seems not to be affected, but slightly better differentiated, strongly suggest advanced degradation of hemicelluloses, leading to an apparent increase in cellulose content. A thermolysis path of degradation seems to contribute, as suggested also by previous research (Chen et al 2014).

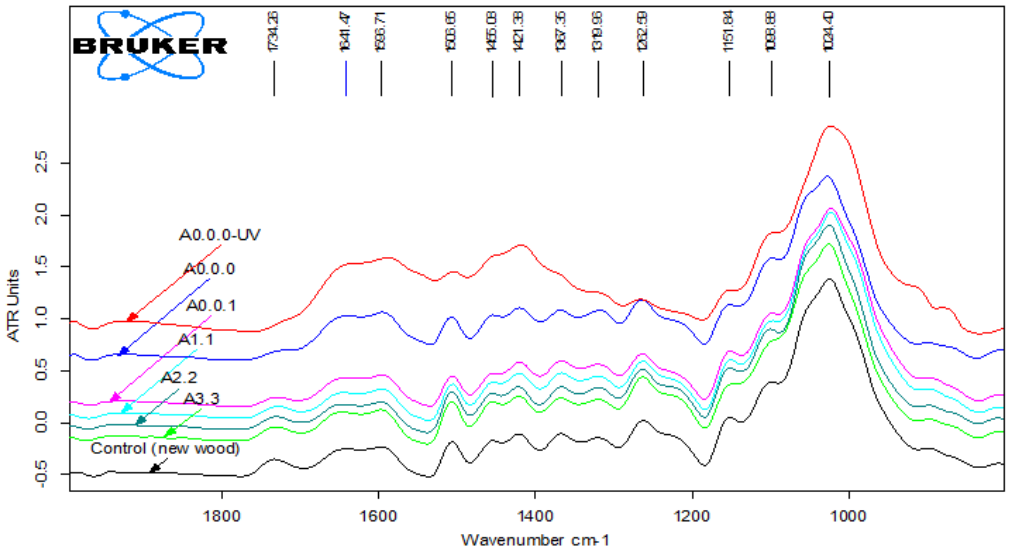

Fig. 4.
Comparative FTIR spectra of new fir wood (control) and aged wood (`283 years), extracted from different layers of the old beam (d. 1733).

UV induced degradation, mostly affecting lignin, is a surface and subsurface phenomenon (Wiliams 2005, Kataoka et al. 2007, Živković et al. 2014, Calienno et al 2014, Varga et al 2017) so that in the spectrum of the sample extracted very close from the surface (A.0.0-0), at about 1mm, the lignin absorption bands look to be not affected and this is, of course, the case for all the samples referring to inner parts of the beam (A 0.1, A 1.1, A2.2, A 3.3). For all these spectra the observable changes are the reduction of the absorptions at 1730 cm^{-1} (assigned to acetyl groups in hemicelloses) and 1370 cm^{-1}, alongside a very slight trend of a better differentiation of the band at around 1640 cm^{-1}, assigned to conjugated carbonyls and quinone chromophore groups. These are typical changes for (temperature induced) wood ageing in indoors conditions and absence of light, which affects mainly hemicelluloses through a multi-step reactions chain, based on thermo-hydrolytic and thermo-oxidative processes, while cellulose content is not affected and condensation of lignin occurs (Esteves et al 2008, Matsuo et al 2011, Kačík et al 2014, Liu *et al*. 2017).

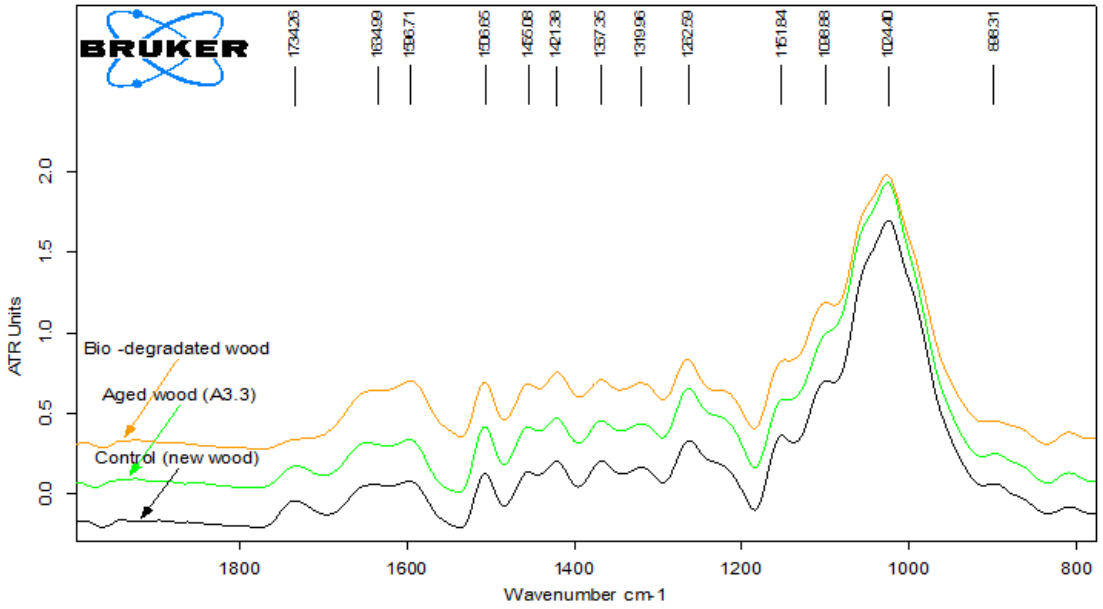

Fig. 5.
Comparative FTIR spectra of new fir wood (control), aged fir wood (283 years, inner layer, without signs of biological attack) and .biodegraded wood extracted from the old beam.

The FTIR spectra in Fig.5 compare detectable chemical changes brought about by ageing (inner sample) and fungal biodegradation, assessed macroscopic as possible brown rot (pocket local area). For the biodegraded sample the band at 1730 cm^{-1} practically disappeared, the bands at 1370 and 1150cm^{-1} slightly decreased, while the lignin absorption bands seem unaffected (1506, 1262 cm^{-1}) or slightly increased (1600 cm^{-1}). These changes are in accordance with the characteristic attack of brown rot fungi, affecting the carbohydrates from the wood cell wall (e.g. Pandey and Pitman 2003, Dobrică et al 2008). Again, hemicelluloses, or at least acetyl groups from their structure, seem to be more rapidly attacked than cellulose.

The data in Table 2, representing the mean values of the integrated areas of some relevant absorption bands, in comparison to the corresponding values for control new fir wood, better highlight and compare the chemical changes brought about by ageing, from the surface of the beam towards the inner layers, up to 100 mm depth, with those determined by brown rot fungal attack.

Table 2

Integrated areas of relevant absorption bands for fir wood (Abies alba* Mill.*): aged and biodegraded wood extracted from the old beam compared to new sound wood (absolute and relative values)

Absorption band (Integrated area)	**Control wood**	**Aged wood**						***Bio-degraded wood*** (brown rot)
		A.0.0.-UV	**A.0.0-0**	**A.0.1**	**A.1.1**	**A.2.2**	**A.3.3**	
Depth from surface,[mm]	*0*	*0*	*1*	*25*	*50*	*75*	*100*	*n.a.*
A 3400	**507.69**	382.40	534.63	526.17	513.95	511.69	551.52	***508.64***
A 1730	**4.71**	0.00	0.56	1.82	2.87	3.05	3.31	***0.00***
A 1506	**6.51**	1.16	5.44	5.47	6.25	7.38	7.68	***6.76***
A 1370	**2.82**	0.44	1.89	2.42	2.63	2.68	2.29	***1.72***
Relative values reported to control								
A 3400	1.00	0.75	1.05	1.04	1.01	1.01	1.09	***1.00***
A 1730	1.00	0.00	0.12	0.39	0.61	0.65	0.70	***0.00***
A 1506	1.00	0.18	0.83	0.84	0.96	1.13	1.18	***1.04***
A 1370	1.00	0.16	0.67	0.86	0.93	0.95	0.81	***0.61***

Variation of density and mechanical properties (bending strength, compression strength) of wood samples extracted from the beam, from the external layer to inner wood (not presented in this paper) are in good relation with the different degrees of ageing and biodegradation revealed by macroscopic examination, colour measurements and FTIR investigation.

Active conservation methods

As previously stated, when considering the re-use of recovered wood for new configurations, the analysis of the initial conservation state and the stabilisation of the material are key issues. Also, the preservation / highlighting of its special aesthetical features, often related to degradation, deterioration or wearing, is important when plastic-expressive qualities of old recovered wood are included in the reconfiguration concept. In contrast, for other cases transformation may include elimination of surface defects and roughness, actually removing the outer degraded layer in order to highlight the colour and features of naturally aged wood.

However, in all these cases some principle operations should be included: adequate cleaning, disinfection (curative treatments against biological agents), consolidation / stabilisation of frail wood, preventive bio protection and surface protective /aesthetical treatments (hydrophobation /coating).

Cleaning of recovered old wood elements generally includes dry (blowing with air, de-dusting with brushes, vacuum-cleaning) and wet treatments (employment of different cleaning solutions). This might be time-consuming and different technical approaches might be considered, from the classical ones to laser cleaning (when available and appropriate), Mechanical processing of surfaces might be applied.

Stopping any active biodegradation processes is crucially important. Physical and chemical methods might be employed, depending on the type of material, its volume, type and extent of degradation and the technical facilities. Whenever available, physical methods based on high or low

temperatures, atmosphere with low /none oxygen content, ultrasounds or gamma radiation, would be preferred to the chemical ones, due to their efficiency and "green" character. Chemical methods, based on biocides, are far more readily available, but the concern of employing toxic chemicals should be considered. The chemicals employed should be of low toxicity to human and eco-friendly. Novel approaches worldwide consider natural products.

Consolidation of frail degraded wood at a level to allow its inclusion in the new configuration is obviously also a critical point. This is usually achieved by impregnation with natural or synthetic polymers, which might be modified with nano-particles for better performance (Tuduce ***et al*** 2012).

As final treatment, application of a natural finishing material, such as oil or wax, is often employed to ensure surface protection and to highlight the beauty of old recovered wood.

For the case study presented in this paper cleaning was achieved by air-blowing, followed by brushing and cleaning wet cleaning. Remedial and preventive treatment against insects attack was achieved employing a deltametryne solution, applied by repeated injections into the galleries. Paraloid B72, as solution of 5% and 10% in ethyl-alcohol/ acetone, as such or modified with 2.5% nano-ZnO, was employed to consolidate local frail areas. An insecto-fungicide impregnation base-coat (based on organic low toxicity biocides with 5% alkyd resin) was applied to ensure preventive protection. Finally, a layer of beeswax (20% in white spirit) was applied and the surface was then gently polished with soft cotton.

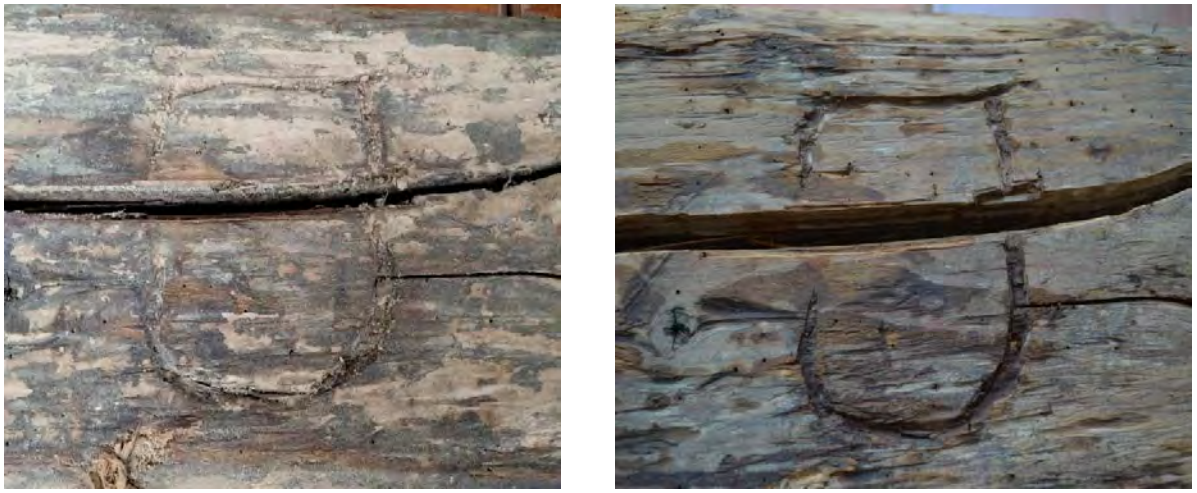

Fig. 6.
Detail before and after cleaning.

These successive steps allowed to highlight the aesthetical features of old wood and to stabilise the material. As a result, the conserved part of this old beam has maintained its historic information and patina, while its aesthetical and suggestive qualities were highlighted. Though of reduced dimensions, this element included in various set-ups of restoration exhibitions in our faculty successfully endured to the visitors the feeling of going back in time, facilitating a better understanding and perception of the restored artefacts in their former original context, which is in fact the main aim of conservation. Of course that very different re-integration ideas could be imagined based on this element and other similar ones.

CONCLUSIONS

A fragment from an old beam from a Saxon house in Ghimbav – Brașov, bearing the inscription *1733 J*, offered the opportunities to study/ investigate degradation and ageing phenomena on old wood elements recovered from constructions.

Colour measurements and investigation of chemical features by FTIR clearly highlighted that ageing phenomena are affecting not only the surface and sub-surface of wood but also the inner part, the effects decreasing from the surface to the centre of the material.

UV radiation is the main factor affecting surface and sub-surface, as demonstrated by the drastically decrease of lignin. Hollocellulose, mainly hemicelluloses are also affected highlighting the contribution of thermolysis phenomena. These are the main phenomena determining ageing of inner parts of wood.

A core of basic principles to be considered in modern design approaches of reinterpretation and reintegration of such elements have been defined. Such a creative process could be understood

as based on three fundamental phases: the existing configuration, transformation and the new configuration.

Initial comprehensive evaluation / investigation of the old wood recovered elements /material, combined with creative design and appropriate active conservation methods may ensure transformation to new configurations with special plastic- expressive impact.

ACKNOWLEDGEMENT

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PROBLEMS AND SOLUTIONS OF CLASSICAL AND INNOVATIVE INTERVENTIONS ON CULTURAL OBJECTS WITH WOOD SUPPORTS

Constantin Ştefan IONESCU
Expert restorer
PhD Student - TRANSILVANIA University in Braşov - Faculty of Wood Engineering
Address/Address: B-dul Eroilor nr. 29, 500036 Brasov, Romania
E-mail: restaurareionescu@gmail.com

Aurel LUNGULEASA
Prof. dr. eng. - TRANSILVANIA University in Brasov - Faculty of Wood Engineering
Address: B-dul Eroilor nr. 29, June 5003 Brasov, Romania
E-mail: lunga@unitbv.ro

Abstract

Consolidation interventions aim at recovering, improving and restoring physical and mechanical properties of wood, a complexed set of actions developed in a technological succession to extend the life of objects that are, in fact, cultural objects, art objects, a national or world cultural heritage, that have been severely affected by physical, chemical and biological degradation.

Our study has taken into account a rich bibliography, including theoretical and practical observations of some specialists from this country and abroad, researches and answers to a series of restoration activities carried out by the author and especially the answers given by these interventions. Our goal is to achieve growth in terms of mechanical strength of the panels affecting the damaged wood, which has losses of anatomical elements (cohesion).

The restoration of patrimony assets implies the approach of techniques and materials in accordance with the state of degradation, the type of artefact, and especially the choice of how to exploit and subsequently expose it. In order for wood to be consolidated, it is necessary to introduce liquid and/or gas materials and/or substances into its microstructure. Transport and penetration into wood is conditioned by a property called "permeability" and the consolidation (the support, the painting layer, or the whole of the work) must be regarded as an important part of the restoration process.

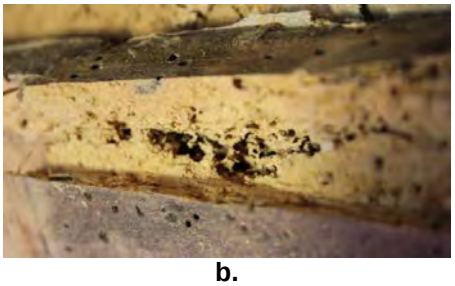
Key words**:** *building; heritage restoration; composite stabilization; conservation; impregnation.*

INTRODUCTION

Wood is a natural material and as such it is predisposed to degradation, especially when insects and fungi come into contact with it, causing significant changes to the basic properties. Damage can be so severe that it endangers the continued existence of the object. Humidity, heat, light, are major degradation factors of the wood structure; also, another factor to consider is the essence of the timber.

Restoring wood means knowing the properties and characteristics, the techniques and technologies that are based on engineering studies of wood. A complementary approach to the two professions is that, in wood engineering, the main objective was to apply treatments and technologies on new healthy wood as standards to improve some properties. Restoration treatments are applied to a wooden panel subjected to biological degradation, with chemical changes, and losses in physical and mechanical properties. In practical terms, on a brittle support, which has reached a powdery state, where the anatomical elements of wood are almost entirely destroyed – Fig. 1 a, b, c, d.

a



b.

c. **d.**

Fig. 1.
Fragmented support
a, b c, d – Massive loss of anatomical elements of wood caused by a xylophage attack.

We recommend the use of science and technology engineering, and transferring their results to improve or restore the structure, the physical and mechanical properties of degraded wood found in support of their cultural heritage assets.

Technical and technological developments allow us today to benefit from advanced technologies, multidisciplinary investigation systems, computer programs or the use of numerically controlled CNC machines.

All these technologies and materials that can be applied to degraded supports are, however, subordinated to regulations and the principles of conservation and restoration of patrimony objects. Therefore, there needs to be a catalogue of materials and established treatments, which yield known results and others, which have less satisfactory results, when their evolution and behaviour are known after the treatments have been performed; the findings and measurements of scientific experts are included in disseminated articles and presentations in restoration conferences – their advantages are shown, but most importantly, the drawbacks of these treatments are elucidated (Wang 1985, Crisci et al. 2010, Ionescu 2016).

OBJECTIVES

The use of compounds close to the anatomical, physicochemical and mechanical structure of the constituent wood in order to allow for: the restoration of the physico-mechanical properties of wood, in our case, affected by the biological attack, the reconstruction of anatomical elements of the support, in order to prolong the life of the patrimony assets.

Increasing the mechanical strength of affected wood panels, which suffered loss of anatomical elements.

The development of a product and / or intervention process on wood support, fragmented by biotic and abiotic factors.

HISTORY

Consolidation interventions aim at recovering, improving and rebuilding the properties to extend the life of objects, which are in fact cultural goods, art objects, a national or world cultural heritage.

Wood consolidation is a complex technological succession; the main purpose of which is partial restoration to achieve, as close as possible the natural state of the physical and mechanical properties of a support that has been severely affected by physical and biological degradation (Ionescu 2016).

A common material used on a large scale by specialists in conservation is **Paraloid B72** consisting of two copolymers - methyl methacrylate and ethyl acrylate, produced by Röhm and Haas in 1935. Paraloid B72 is a thermoplastic resin soluble in acetone, ethanol, toluene, xylene, ethyl acetate, butyl etc.

In our research, we found that strengthening interventions considered as "manual", like injection, dripping, brushing, remain only at surface level or shallow in terms of depth of penetration into the wood cells, filling is relatively low. In order to bring about an obvious effect in improving the mechanical strength, injection or brushing must be carried out in a considerable number of repetitions and the viscosity of the solution and the type of solvent are very important.

It is known that in hardwood, because of the complex structure "fluid flow is more difficult" (Siau et al.1984); permeability is an important factor affecting the retention of liquid in comparison with the pressure and viscosity. Intercellular voids in the wood structure will result in retention of treatment solutions.

The polarity of the solvent stream affects the penetrating flow. The wood is more permeable to non-polar than to polar solvents (Wang Schniewind 1985). Non-polar viscosity allows deep penetration, and high polarity solutions cause swelling in wood.

After evaporation of the solvent, the polymers reinforce the structure. The most common treatment used to strengthen the wood are acrylic polymers (Paraloid B72). The solvent chosen must be appropriate to: dissolve the polymer, penetrate into the wood, and to not cause colour reactions with components of wood and deterioration of pictorial layers. Another important parameter is the degree of swelling of the wood, as a result of interaction with the solvent. Also, the choice of solvent, toxicity, explosiveness and flammability must all be taken into consideration (Aurerson 2000 cited by Mankovski 2015). For example, according to a study by Paciorek (1993) the greatest degree of saturation of the timber was obtained by using Paraloid dissolved in methanol, however, due to swelling, it could not be applied for practical conservation. Therefore, the most commonly used solvent is toluene. Paraloid solution B72 with a low polymer concentration penetrates the wood best, but for efficiency it requires repeated impregnation. High saturation causes a reduced penetration of the solvent in the depth of the timber (Schniewind 1990, Wang and Schniewind 1985, Schniewind and Eastman 1994). In their research, determined the content of polymer in the samples of damaged wood impregnated with 20% Paraloid B72 in toluene, and it was found that there is polymer at a depth of less than 7 mm, in about 10% of the timber vessels. Better supersaturation was obtained by dissolving the Paraloid in acetone (Mankowski et al. 2015). Acetone causes dimensional instability of the wood and its use in the restoration should be judiciously observed.

MATERIALS, PROPERTIES AND USES

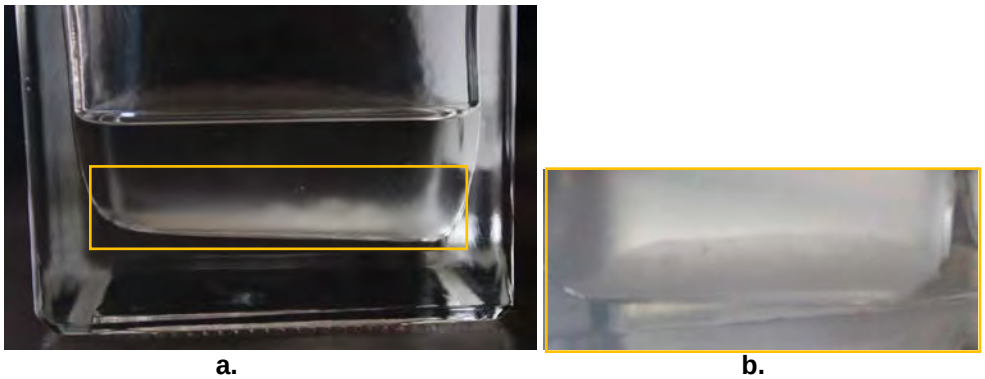
Strengthening damaged wood by impregnation with soluble thermoplastic resin is considered the most promising method because of the physical and mechanical properties, as well as the reversibility compared with thermosetting synthetic resins (Wang and Schniewind 1985). In fact, Timar (2011), specifies that the original synthetic polymer solvent that was set in the timber remains soluble.

The degree of impregnation will depend on: the building material, the solvent used, the concentration and the viscosity of the solution, the permeability of the wood material to be reinforced, the technique used (brush, injection, immersion, vacuum impregnation, etc.) and other treatment parameters such as time and temperature (Wang and Schniewind 1985, Unger and Unger 1994, Unger et al. 2001, Timar 2003, Formakalidis 2006, Timar 2010).

Higher concentration solutions store more resin and will give more resistance. However, for practical applications, where large objects or low permeability panels have to be consolidated, full penetration is not easy to achieve. Absorption can be increased either by increasing the pressure difference, applying positive pressure after vacuum treatment (which would require more elaborate equipment), or by reducing the viscosity of the treatment solution. The latter can also be achieved by reducing the concentration, which is to some extent counterproductive, as it reduces retention. The choice of solvent and the concentration becomes one, which cannot be done in absolute terms, but must be adapted to the particular requirements and conditions of the subject to be treated (Schniewind and Wang, 1985).

A similar conclusion we obtained in our *"Scientific study about the determination of masses of active substance needed for treatment of wooden panels degraded by xylophage attack"* (Ionescu 2016*)* where we presented measurements and determinations of their masses, of the solvent, of Paraloid B72 at various concentrations and the panel on which it occurred. The parameter measured was the necessary and acceptable quantity of non-volatile, active substance required to strengthen the restored panel. Over the course of 3 years, we started a study that took into account the required amounts of the builder (Paraloid B72) and the solvent type with different polarities from non-polar (e.g. Toluene, Xylene) to medium polarity (ethyl acetate, butyl acetate, acetone), which have a different volatility. Our findings, as in accordance with those of Schniewind (1990) and Mankowski (2015) tell us that solutions with high polarity can cause swelling of the wood and the penetration depth of solutions with high viscosity is more difficult.

Another material used for restoration, is **Regalrez 1126** - a saturated cyclic hydrocarbon similar to wax and paraffin (Crisci et al. 2010). According to the tests made by us, it was found that the mixture of the two substances simultaneously (Regalrez 1126 and Paraloid B72) can produce a crystallization, resulting in a suspension, which makes it difficult for injection, that could lead to a negative effect on the expected outcome. It is recommended that the steps are to be done differently. Our conclusion was the application of the two independent substances, at an interval of drying of at least 8 hours (Fig. 2 a, b).



a. b.

Fig. 2.
Particles in suspension
a. deposits; b. detail.

The efficiency of a consolidation treatment will ultimately depend on the amount of consolidate remaining in the material/object (consolidate retention), the depth of penetration and the uniformity of its distribution, all of which can be aggregated under the term of degree of impregnation (Timar et al. 2011), which is otherwise found new in these techniques.

Taking into account the concentration and the solvent volatility, permeability is reduced the more the anatomical elements of wood are degraded by a xylophages and fungal attack, and where flight holes are clogged, blocked by the wood sawdust produced by chewing insects.

In general, the permeability is higher in the heartwood than the sapwood, and it is also much higher than that in the longitudinal direction of the crossbar. Permeability is the most important factor affecting retention, compared to pressure and viscosity (Wang and Schniewind 1985).

TREATMENT, METHODS, EFFICIENCY

Where we have shown that the effectiveness of the treatments by injection, brushing or dipping (where possible) can be reduced by many factors, then, to increase the effectiveness of the treatment, it must be repeated. A much more effective measure is vacuum-pressure consolidation. A clear example, in addition to the one shown in our study (Table 1, 2), is shown in the study of Golez et al. (n. d.), where a panel of wood before the impregnation weighs 1000 g, and immediately after impregnation weighs 2380 g. After conditioning and stabilizing, the weighed object reaches 1370 g. The increase in weight was due to impregnation with the consolidate solution. By using gravimetric methods in our researches carried out during the years 2014-2016, so far, on a number of 24 wooden objects, we made determinations of the fragments entered in the restoration, compared to the mass of new panels (determination with conventional masses of new wood), the mass of solvents and the active substance (Paraloid B72), the mass of the impregnated wood, at various stages and with various concentrations, and then the final mass of the restored object. Our study shows that the masses of panels were determined before, during, and after the final stabilization, and quantities were found that each time approached the mass of the panel on which it had intervened or even surpassed, and that at the end of the intervention, the remaining mass, fluctuated between 18.4% and 60%.

Table 1

Determination of masses of residual substance required for the consolidation of wooden panels

Name of the icon Inv. No. Essence	**Mass stage 1**	**Stage 3 final mass**	**Remaining substance mass**	**Required mass for consolidating (percent)**
Baptism of the Lord 761 walnut panel	882	1095	213	24.15%
The welcoming 762 lime panel	638	806	168	26.33%
Dying of the Saint Mother 763 walnut panel	521	835	314	60.27%
The Transfiguration	732	934	202	27.59%

Name of the icon Inv. No. Essence	Mass stage 1	Stage 3 final mass	Remaining substance mass	Required mass for consolidating (percent)
766 lime panel				
Descent to Hell 774 lime panel	216	533	317	46.76%

Table 2

Mass determination during restorations

Inv. No.	Initial dimens-ions (mm)	Healthy wood mass (g)	Panel mass T0	Mass after excavation	Consolidation stage I B72.	Stage II B72 12%	Stage III B72 20%	Final mass after approx. 60 days
760	415 x 345	1889	1380	-	1412	1479	1510	1477
764	415 x 345	1889	1270	-	1753	1848	1968	1748
768	332 x 240	1051	738	573	831 Adding td. & c. B72 6+12%	949	880	770
771	320 x 270	1140	970	846	1146 Adding td. & c. B72 6+12%	1243	1278	1001
775	320 x 120	506	277	161	351 Adding td. & c. B72 6+12%	379	426	388

EXPERIMENTAL STUDIES, DISSEMINATION

We believe that the efforts of internationally recognized specialists, chemists, biologists, physicists, engineers, restorers, teachers, etc., who have valuable contributions to research and dissemination, and extensive and beneficial results in the field of conservation and restoration of heritage objects, must be further analysed and applied to our own research, in order to improve the techniques and materials used for the salvation of works of art.

When substances and solutions introduced into wood do not have the expected effect, when the mechanical strength of the support is diminished, and the valuable painting layer is in danger of irreparable loss, practical radical interventions called "prosthesis" are imposed (Ionescu 2014).

The structural and dimensional restorations are made through the use of fragments of wood, from the back to the front painting, to create a structure for stability by removing the strongly degraded base, and the panel then geometrized with the new wood elements, so that the contractions and swelling of the wood produces minimal impact on the painting layer, but also on the support in general (Ionescu 2014).

Composite materials resulting from the stratification of wooden planks as the horizontal axis and the vertical thickness, can be addressed as treatments and techniques to restore wood by the very fact of the arrangement of the blades, taking into account anisotropy and the tendency for natural bending, to form both the width and especially the thickness of the alternating layers. However, the joining lines of the materials must not be overlapped, in order to reduce internal stress and dimensional variations (Ionescu 2016). Our research has shown that this way of putting into practice some of the fragments required for addition, arranged in such a method, have resulted in the development of practical techniques. The old and degraded wood as well as the new wood brought in as additions, results in a more stable material with low dimensional variations, which gives the painting increased stability; tensions caused by wood play are reduced. We can find and practice with new materials, plywood, etc., but only where we do not have layers perpendicular or angled fins attached.

Following numerous interventions of restoration, the support showed significant deterioration, cracking, fracturing, detachment, or worse, where degradation led to great losses; this guided us to develop concepts for interventions to restore on the traces of fracturing by additions (Fig 3. a, b, c) when the fracture and loss of support of the painting layer was between the two areas.

a. **b.** **c.**

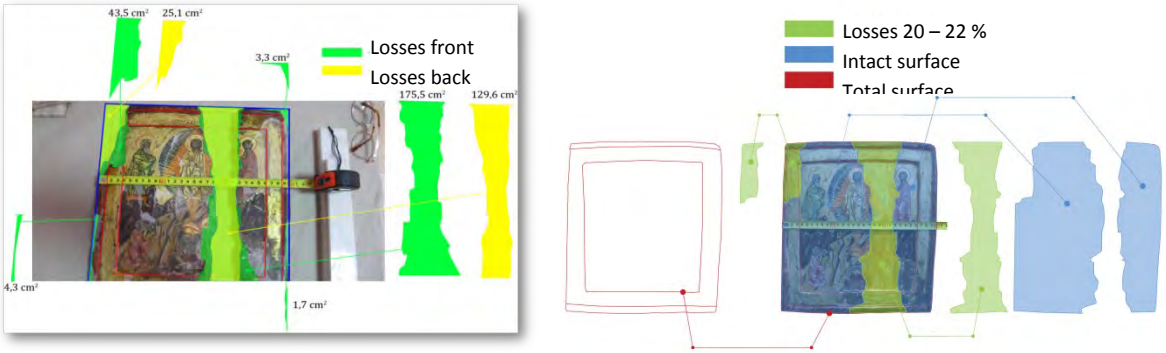
Fig. 3.
Icon 766
a. before the restoration; b. back before reloading; c. after restoration.

Observing the fractures from the pictorial layer, on the one hand, and from the back, on the other hand, it can be seen that they are not in the same plane, and the shape is irregular due to fracturing and loss. Our restoration intervention was carried out by adding composite material (wood) for the reduction or cancellation of the panel tensions.

On the front, there is a paint layer, which required special attention, due to the fact that the cradle was to be merged into the particularly rundown support caused by the xylophages attack. The painting showed evidence of fracturing, this imposed making tracings of the irregular contours, the two fragments were detached, and the intermediate piece was to take the shape of the fracture so as not to press the edges of the paint layer, which showed variable thickness between 0.3-2.1 mm., these measurements representing the paint layer, primer and the rest of the wood. Basically, it was not enough to copy the 2D fracture track, but it required a 3D approach.

Note that it is possible that this panel was originally constituted from two panels, although the reduced panel dimensions (about 267 x 335 mm) did not require this, especially since the similar icons of the same group were made up of a single slab.

Our hypothesis on this type of degradation and fracture, with detachment and fragmentary loss of the median area, was generated by the very probable sticking together of two wood floors which, with aging adhesive and detachment, continued the progressive degradation of both fragments, causing the loss of important areas. If the panel surface constituent is about 894,4 cm^2, then the losses on the painted surface measure approximately 228.3 cm^2, and losses to the reverse side approximately 183.1 cm^2 (Fig 4 a, b).



a. ***b.***

Fig. 4.
Graphical representation of support losses
a. Differential marking of loss seen from the front and from the back
b. Graphic representation of additions - front view.

RECONSTRUCTION BY FOLLOWING THE LINE OF FRACTURE

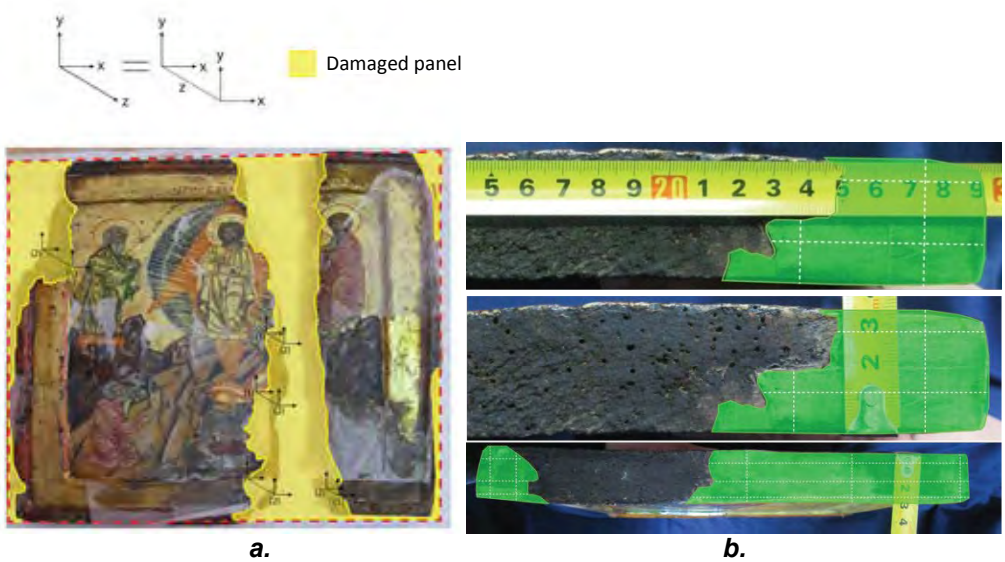
This intervention has generated a series of researches and studies, which have been crowned with recording and registering with OSIM (Office of Patents and Trades) of a "Method of consolidation - restoration and monitoring of wooden heritage objects" (Ionescu, Lunguleasa 2017).

The invention (Ionescu, Lunguleasa 2017) relates to a process for the restoration and protection of national and international heritage objects with wood supports. In its realization, it starts from the observation and determination of the conservation status, with the loss of the physical-mechanical properties, of the wooden support, of the carved and/or painted polychrome wood, of the patrimony objects that are undergoing the restoration intervention. Also, the present invention is used to limit the risk of falsification, theft, illegal exports, paintings, icons, etc., which can easily be transported or replaced, or by changing the identity of the owner.

The problem solved by the invention is to provide 1. a method of restoring the wood substrate sheet that have areas with a high degree of degradation, loss of support and fracturing, and 2. protecting them against theft or forgery.

1. The restoration of the wood substrate sheet objects by reconstituting traces of the fracturing of the wood substrate, where the sheet objects are in a state of degradation (Fig. 5 a, b):

a. Reconstruction is done by physical and mathematical determinations of gauge dimensions.
b. By comparative measurements of the object itself or similar objects belonging to the same production, time, author, etc.
c. By recomposing the drawing represented by the iconographic theme or the scene.
d. 3D tomographic scan of the fractured detached object and loss.
e. Transfer of the imaging result into a numerical control machine (CNC) program.
f. Making the missing piece on the machine in coordinates, on the traces of fracture, and incorporating a three-dimensional prosthesis, to reconstitute the shape and the gauge.
g. Bonding of old and new components with vinyl polyacetate materials that are reversible.
h. Showing the geometry of the patrimony object with respect to the flatness or curves in the cutting plans to which the heritage object was subjected in its life cycle, from the origins to the moment of the interventions.



a. ***b.***

Fig. 5.
Reconstitution on fractures.

2. Protecting and monitoring of heritage objects by implementing an identification chip.

One of the main advantages of the current invention resulted from implanting heritage objects of particular value with an identification chip, which are then ranked in a legal form, fund or treasury. The implementation of a microchip on patrimony objects is done to limit the risk of theft mainly by illegal exports of paintings, icons, etc., which can easily be transported, changing the identity or the owner. The procedure consists of the implantation in a certain area, which is well determined and known at national and international level by specialists from the competent institutions, who have the obligation and possibility to easily check, research, scan or identify patrimony objects that have the general characteristics of art objects. This application may be for the benefit of collectors, auction houses, the customs, etc., and allows easy identification of stolen or incorrectly catalogued items. The chip is loaded with the identification data of the patrimony object, the represented theme, owner, date, year of

restoration, style, etc., to conserve the authenticity, and other interventions that are governed by the principles of restoration and specialized legislation in force.

CONCLUSION

In conclusion, restoration interventions include the integration and use of classical or innovative materials or techniques and is a long-term process, whose results are quantifiable over long periods of time, precisely by determining the influences or behaviour of materials both individually as well as in the combination of interventions. Therefore, we note that the dissemination of studies, the implementation or the development of researches by restorers, based on extensive and valuable bibliographic documentation, may be the basis for superior restoration processes that will benefit the patrimony objects in order to recover and save objects of art and of national or world cultural values. The purpose of using new, advanced techniques and state-of-the-art materials, is subordinated to limiting or stopping degradation, restoring or re-establishing the lost physical and mechanical properties for art objects on wooden panels, that have been degraded over the centuries, but surprising, whose pictorial layer of a special value, was kept inversely proportional to the support losses. That is why the knowledge of materials and their behavior over time is necessary, not only at the level of workshops or schools, but all the more so at the international level, through the understanding and application of materials and solutions that were produced by the centres or laboratories of cultural, European or world institutions, whose best practices are based on research of interdisciplinary high-level scientific laboratories.

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SECTION 7. FURNITURE DESIGN

WOVEN FURNITURE DESIGN: IN SEARCH OF FORM AND TEXTURE

Regina RAYCHEVA
University of Forestry, Faculty of Forestry Production
10, Kliment Ochridski Blvd., 1797, Sofia, Bulgaria
Tel/Fax: +3592 962 4198, E-mail: rexi@abv.bg

Dessislava ANGELOVA
University of Forestry, Faculty of Forestry Production
10, Kliment Ochridski Blvd., 1797, Sofia, Bulgaria
E-mail: d_spasova@abv.bg

Abstract

The paper researches wicker furniture design in an attempt to analyse the existing materials, techniques, tradition in basket-weaving and furniture-weaving. This leads us to research into the potential of wicker in form-building and provides an explanation of modern designers' interest of in this material and technique. The analysis is aiming at revealing the bi-fold role of the weaving technique both as a surface-covering and as a structural building material. In this way, the shift from the simple use of weaving as caning, to structural building of objects, is outlined. The authors attempt a morphologic analysis of woven furniture examples, structural classification according to types and kinds of materials used; this is done by case-studies of 20 examples, selected for their interest and archetypal view, as well as their noted authors. In this way, we can give an answer to the question: Is woven furniture strictly kept into the realm of outdoor and hotel use, or can it 'return' to our homes with the corresponding functional and aesthetic potential?

Key words: *woven furniture design; wicker; basketry techniques; rattan furniture; contemporary design.*

INTRODUCTION

The Term Wicker

As part of the theme of crafts and modern design, in this paper we shall attempt to see how the 'crafty' look of woven furniture is relating to serial production; and why it is so attractive to modern man. The Webster's Dictionary (1986) gives the following explanation of the word 'wicker': "a small pliant twig or osier: a rod for plaiting basketwork; 2a: wickerwork, b. something made of wicker (as a basket)". According to Miriam Plans (2004), "the word 'wicker' is believed to be of Scandinavian origin, coming from the words 'wika', which means 'to bend' in Swedish, and 'vikker' meaning 'willow'. Many people think that 'wicker' is an actual material. Rather, it is a class of furniture woven from a number of materials, including rattan, cane, bamboo, reed and willow". The materials, from which all kinds of woven products are made, are a great number. Here we can enumerate osiers, sea grass, sweet grass, palm leaves (to give some tropical examples), raffia; but also the twigs of all sorts of local plants, e.g. the vines of blackberry, etc; to which we can also add the needles of the American Southern Longleaf Pine.

OBJECTIVE AND METHOD

In this paper, the authors use a case study method. Selection of individual examples was done both for their originality and the specific type they represent. On their basis, selection, classification, visual identification and comparative morphologic analysis was used to bring out the connection between basketry and furniture caning and weaving. Tables were used to present graphic material in an orderly manner. Our paper uses research of material, classification of basket types and derivative sculpture objects, mat-weaving patterns; this section is aimed at discovering more sources of design inspiration. Case studies of modern wicker furniture design are researched, on the basis of which we outline features, morphology and, most of all, form-building potential of wicker. To bring clarity in a visual matter such as design cases, the authors have researched design trends developed in time. The paper is aimed at becoming a source helping designer research.

EXPOSE
MATERIALS

A great number of natural and man-made materials are used for making woven products, they provide strips weaving. A brief overview of some of these materials follows.

NATURAL MATERIALS

The most common natural material used for the production of woven furniture is **rattan**. Roughly 600 species of palms in the *Calamoideae subfamily* exist most of which are "climbers that use thorny stems and leaves to hold on to the supporting structure of other plant species" (Meijaard et al. 2014). Rattans have slender stems, 2–7 cm diameter and long internodes between the leaves. With a growth rate of 36 inches per day, it is an abundant and renewable material. Rattans are cultivated within forests and swidden land. "Rattans are ready for harvest 5–7 years after planting. In clustering species, the general harvesting cycle is 3–4 years, with each cluster of rattan stems yielding around 20–25 kg" (Meijaard et al. 2014). Because of its flexibility and its long stems of great strength, rattan is primarily a building material. In its un-split form, it is used to provide structural parts in furniture and construction. "The outside skin of the rattan pole is usually peeled off, to be used as rattan weaving material. The skin is cut into strips used for the weaving process of seats and backs for chairs, and also to bind furniture joints together for reinforcing and decorating purposes" (Benhua et al. 2016).

Bamboo, a member of the grass family *Gramineae Poaceae*, includes over 70 genera and 1200 species. Most bamboo species are native to warm and moist tropical and warm temperate climates. "One of the fastest-growing plants in the world, it can increase to 10-30 m in 40-60 days, and reach complete height and diameter within one season. Bamboo usually can reach optimum material properties for 4 years; its timber has special properties of split, easy preparation, high strength, moderate rigidity, and good toughness, much higher than that of general timber" (Benhua et al. 2016).

"The wood properties of bamboo mainly depend on the components and structure of the cell wall. (…) Like wood and agriculture residue, bamboo is mainly composed of cellulose, hemicelluloses, and lignin, even though the contents of these compositions are different"(Benhua et al. 2016). As an important biomass material, bamboo could be sustainable for utilization once planted. "Bambusa vulgaris is mostly cultivated by the rural community because of the high growing rate, thick culm wall, uniform internode size and the high yield of shoots produced. 2 year-old culms are used in handicraft and basketry industry; 4 year-old culms are normally used for panels, parquets, furniture and construction purposes" (Wahab et al. 2009).

Bamboo stems are processed by splitting into halves, quarters or eighths. Having high elasticity, the material can be bent after harvesting, and is dried bent (Бърдаров Н, Владимирова Е, 2014). Like rattan, bamboo has a hard outer shell, which is peeled, in order to make a weave. It is also used for the structure of wicker furniture. Its natural durability of less than two years is due to high levels of starch. These turn untreated bamboo to material easily vulnerable to fungi, rot and attracting insects such as termites (Boran et al. 2013).

Palm leaves are used to make woven products such as bags, carpets and furniture. Abacá, also known as Manila hemp, is a species of banana (*Musa textilis*), native to the Philippines, grown as a commercial crop in the Philippines, Ecuador, and Costa Rica. The plant grows to 4.0–6.7 m, and averages about 3.7 m. The fibre, extracted from leaf stems, is classified as hard fibre, along with coir, henequen and sisal. The leaf sheaths contain the valuable fibre (Bailey 1947).

The Raffia palms (*Raphia*) are a genus of about twenty species of palms native to tropical regions of Africa, and especially Madagascar, with one species (*R. taedigera*) also occurring in Central and South America. They are remarkable for their compound pinnate leaves, the longest in the plant kingdom; leaves of *R. regalis* up to 25 m long and 3 m wide are known (Tucker et al. 2010).

Cane (the plant) is any of various tall, perennial grasses with flexible, woody stalks, and more specifically from the genus *Arundinaria* (Brako et al. 1995). Cane is used "… for weaving baskets, for hampers, chairs with the use of seagrass to beautify it, for beds of different sizes for children and adult, cupboards, tables of different shapes and sizes and can also be used for walking sticks. It can also be used for boats and roofs according to history" (wikipedia.org). Caning (furniture) is actually the weaving of chair backs and seats with the outer peeled rattan skin. Cane is also used in combination with structural rattan materials for surfacing furniture (Day 1917).

Cattail is the material used in genuine rush weaving and is the common marshland plant found almost everywhere in northern latitudes. These plants have many common names: bulrush, reedmace, cattail, punks, corn dog grass, etc. These are names of plants of the genus *Typha* – plants in the family *Typhaceae* (Stevens 2013). The leaves of the cattail are harvested, dried, pressed flat, twisted together two to five strands at a time to make various widths, and woven in some variation

of the diagonal cross pattern. Cattail is woven onto frame that forms the seat and back of the chair. This material is very durable with beautifully variegated coloring, featuring hues of pale greens and ambers. With time cattail mellows into an equally attractive golden brown (furniturerenewal.com).

Seagrass is a flowering plant, belonging to four families, all in the order *Alismatales*, which grow in marine, fully saline environments (Waycott et al. 2014). It is used in baskets and furniture, and woven like rattan. This grass comes in varying strand sizes and can also vary in the tightness and uniformity of the twist.

Hierochloe odorata or ***Anthoxanthum nitens,*** also called Sweet Grass, Manna Grass, Mary's Grass, or Vanilla Grass, etc., depending on the geographic latitude. It is a plant which is common above 40 degrees north latitude in Asia, Europe, and North America. (Hope and Gray 2009). "The plant is harvested by cutting grass in early to late summer at the desired length. Sweet grass is sun-dried and must be soaked in warm water until it becomes pliable. The pliable grass is typically braided into thick threads and then re-dried for use" (wikipedia.org).

Different tree species are also used in view of their unique qualities: wood flexibility and easy splitting. Such is the case of **black ash** (*Fraxinus nigra)* in the USA. Since it does not have fibers connecting the growth rings to each other, it can be easily turned into splints. Other species include the willow, osiers and hazel switches, birch bark, poplar, etc. Tree roots, e.g. the roots of spruce, fir, cedar are known to be flexible and of great length, and are gathered for weaving as well. **Willow** is a natural material from Europe and the United States and is a common weaving material that comes from willow trees and shrubs of the genus *Salix,* family *Salicaceae*, mostly native to north temperate areas and valued as a species grown for its decorative qualities, providing shade, erosion control, and timber.

Vines of various local landraces (bramble, or blackberry – *Rubus fruticosus*) are used for basket-making; corn leaves and straw are other materials.

Pine needles of *Pinus palustris*, commonly known as **longleaf pine** are used for a specific coiling technique used for baskets. This species is native to southeastern United States (Farjon 2013). The average length of the longleaf pine needles is 15 to 38 centimetres. The ancient craft of coiled basket making was conceived by Native Americans in Pre-Columbian times.

MAN-MADE MATERIALS

Paper-wrapped high tensile wire is made from heavy kraft paper twisted to form uniform strands and formed into large coils. The weaving of paper-wrapped high tensile wire was invented in 1907 by the American Marshall B. Lloyd. This machine-woven fabric became known as Lloyd Loom and it revolutionised an area of the furniture industry. "In 1921 Lusty, a packing case manufacturer, acquired the rights to mass-produce furniture using the American method of weaving twisted paper fibre, patented under the name Lloyd Loom. The product, which could be woven in a variety of patterns, was attached over bentwood frames and often imitated popular furniture forms made in other materials" (Williams 1994).

Synthetic materials

Synthetic wicker furniture is usually made of aluminum structural frames. Resin-wicker refers to synthetic material, usually **nylon**, **polyethylene**, **high-density polyethylene** *(HDPE)*, **vinyl** or **PVC**. Woven plastic strips cover furniture frames made of metal or wood, and are much more durable than traditional wicker (patioproductions.com). Modern synthetic materials used in furniture are UV resistant and weather-proof, non-toxic and non-pollutant; pleasant to the touch, low-maintenance, lightweight and long lasting.

USES: BASKETRY AND OTHER OBJECTS

There is an obviously analogous manner of building baskets and furniture pieces. Basketry includes the creation of "receptacles made of interwoven, rather rigid material, but it may also include pliant sacks made of a mesh indistinguishable from netting – or garments or pieces of furniture made of the same materials and using the same processes as classical basket-making" (Balfet 2015). In this respect, the same author goes on to say, that this "handmade assemblage of vegetable fibers … is relatively large and rigid, so as to make a continuous surface, usually (but not exclusively) a receptacle".

Hélène Balfet (2015) distinguishes two basic structural types of baskets: coiled and non-coiled constructions. The first predominate "in dry, subtropical savanna regions or roots and stalks found in cold temperate zones". Non-coiled are subdivided into three types: wattle construction, lattice construction and matting or plaited construction. Plaiting techniques are used in tropical zones that "have palms and large leaves that require plaiting techniques". This explains "sewed coiling that

predominates in the African savannas and arid zones of southern Eurasia" or "coiled construction", as the author calls it, against "various forms of plaiting in hot regions" – or "wattle" and "lattice" constructions. The wattle construction represents "a single layer of rigid, passive, parallel standards held together by flexible threads in one or three ways, each representing a different type" (Balfet 2015). Matting construction of weave is different, because "standards and threads are indistinguishable in matting or plaited construction; they are either parallel/perpendicular to the edge (straight basketry) or oblique (diagonal basketry). Such basketry is closest to textile weaving" (Balfet 2015). Similarly, the British basket-maker Polly Pollock (1993) differentiates between four different basket-weaving techniques (Table 1): **coiling technique** ("stitching and wrapping … bundles of grass, the basket spirals up"); **plaiting,** a technique using strips of materials either to form a bias weave (45°) or checker weave (90°). No distinction is made between warp and weft in this type. **Stake and strand** technique means that the baskets feature structural stakes, woven with a softer and flexible strand, or weaver. Cane and bamboo can be used for the structural stakes in Asia, while willow is the material used in Europe. Polly Pollock points out a fourth technique, namely **twining**, which is also using warp and weft elements.

Table 1.

Types of baskets

			
Coiled	Stake and Strand	Plaited	Twined

Table 2.

Types of basket weaves

			
1. CHECKERED WEAVE	2.DIAGONAL TWILLED WEAVE	3.VIENNA CANE	4.TRIAXIAL WEAVE
			
5.TRI-AXIAL (MAD) WEAVE	6.BASKET WEAVE	7. TWILLED WEAVE	8. 'NON-WOVEN' TYPE
			
9. TWINED WEAVE	10. CHECKERED OPENWORK WEAVE	11. OPENWORK TWINED WEAVE	12. OPENWORK TWINED WEAVE

One of the most popular patterns is the "Vienna Straw Weave", which combines perpendicular and diagonal weaves at the same time.Stuart Lawson (2013) refers to rattan weave as 'structural textile': "Woven textiles can be flexible or rigid and use thread, encapsulated wire or various organic fibres such as rattan for their construction. The more rigid the strands, the less need there is for tension or framing to support the weave. However, thin-stranded, open weaves such as a cane-woven

seat, work very well under tension to provide elastic, comfortable cushioning with free air movement, using a minimum of materials. Nearly all structural textiles applied to furniture are industrially produced, pre-woven sheet products that, with varying degrees of handwork, are tension-fitted to frames. A smaller-sized craft industry weaves and applies single strands to furniture by hand" (Lee 2011).

Basket-weaving has been raised to the level of sculpture art by modern Japanese authors. "Japanese ikebana baskets still echoed the Chinese archetype, which maintained a vessel form that served to contain the flowers" (Lee 2011). The exhibits from the exposition "Japanese Baskets and Sculpture in the Cotsen Collection", held at Asian Art Museum, San Francisco, in May 2007, show a remarkable richness of structural weaving methods. "The techniques of weaving bamboo in strips vary with each basket (…) Many of the baskets were originally made for the tea ceremony or for flower arranging, activities with profound artistic and philosophical meanings in Japanese culture. And many were created by artists who represent basket-making lineages and by others who have been designated in Japan as "Living National Treasures" in recognition of their mastery" (The Asian Art Museum 2007). The transition from a functional object (tea ceremony flower basket) to abstract sculpture is a proof that authors, aware of the form-building potential of the weaving technique, naturally evolved to abstracts. This gives us the ground to find a potential to turn those structures into designed furniture pieces.

WOVEN STRUCTURES: SHORT HISTORY

A great many number of household items are made by weaving fibres of different plants. Such objects have been found in Ancient Egyptian tombs, or have been depicted on Greek vases, on wall paintings, carved out on gravestones etc. Many objects were prepared by organic fibers: such as woven rush mats, baskets, sandals, trays, chests, wickerwork stands, conical hats in Asia, tea strainers in Japan, etc. In Ancient Rome, round wicker armchairs were produced, considered to be originally made by the Etruscans. Later (17^{th} C), caning began to be used to substitute costly upholstery fabric. The 19^{th} century was particularly abundant in exotic vogues, and caning was widely used for Thonet chairs, Victorian extravagantly decorative wicker chairs, etc. The beginning of the 20^{th} century saw the emergence of the Lloyd loom technology of Kraft paper used to machine-produced textile for garden chairs. The use of woven furniture for hotels, restaurants and gardens dates from this time. The second half of the 20^{th} C saw the rise of interest in rattan furniture as decorative characteristic pieces in residential interiors.

WOVEN FURNITURE: CLASSIFICATION BY FUNCTION, STRUCTURE, MATERIAL AND FORM-BUILDING

Woven furniture use today ranges from garden furniture, contract furniture for restaurants, resort hotels etc., to characteristic single residential furniture pieces, such as chaise longs, garden sofas, rocking chairs, swing/hanging chairs, children's chairs, coffee tables, book étagères, coat hangers, flower stands. The materials used differ widely: wicker furniture wholly made of rattan (structural poles and 'skin'); metal or wood structure plus caned parts; or structure of rattan or wood plus Lloyd Loom woven panels; metal or wood structure plus other types of mesh (woven tubular knit, leather strips, polyethylene strips, felt, laser-cut leather, etc.). The techniques are widely used for other home textiles, such as rugs, wall decoration, lighting fixtures, etc.

Woven furniture follows basically the rules of basket weaving; therefore some of the shapes thus produced have a definite likeness to that of a basket, or receptacle; here we can quote Palla Chair, Primavera Armchair, E10 Rattan Chair, etc. A creative manufacturer, such as Paola Lenti, uses the African basket-weaving coil technique in some of its new models ("Afra"). Woven chairs are characterized by metal/rattan/wood structure, and a structural weave, usually tight, with some exceptions. The pieces are sculptural and generally consist of one single part or a small number of parts, involve exclusive hand work, and have a great visual effect. Here we have found the following morphologic typologies: 1. "Closed Spherical Type". This is closer to a container than a proper frame furniture structure, morphologically it forms closed volumes (Fig. 1, Table 7). Here belong other closed shapes, such as Moebius Strip, ovoid shapes etc. 2. "Open Basket Type" (Fig. 2, Table 3). 3. "Flower Basket Type" (Fig. 3). This is a conical type, typical for a basket container, with a very decorative effect and a clear narrowing under the seat and wide flaring backrest. 4. "Classical Roman Type" (Fig. 4, Table 4). Starting from a 'basket' type, these chairs feature characteristic half-round plan, ending in a round-shaped backrest with sloping armrests. This type was manufactured by Lloyd Loom in the USA and Britain. In its essence, this is a round chair with a woven surface and 'skirt' under the seat, and is typical for garden furniture use. 5. "Flying Carpet Type" (Fig. 5, Table 5). This type represents a woven metal frame bent in space so as to serve as a structure for both backrest, seat and legs.

Classic example is a chaise-longue "809" by Mario Bonacina. Many chairs feature this concept of woven metal frames for their back-and-seat; here the form-building does not stem out from the weave or rattan pole shape; it is totally subjected to the metal structure. 6. "The Tubular Type" (Fig. 6, Table 3): This type is characterized by using hollow 'tube' shapes all along their length, in order to achieve the seating-cum-backrest surfaces. The 'tube' is also open in order to achieve a lighter look. An example is the Wicker Chair by Marc Newson. 7. „The Mesh Type". Examples, such as Nautica hanging chair, Doeloe lounge chair, feature structures made only of rattan poles. Surfaces are built by thinner stems that are fixed near to each other; or by a mesh made out of such stems. With these two types, the volume is delineated by separate contours. This technique is used both in rattan material and in thin wooden parts.

Fig. 1.
Closed Spherical Type.

Fig. 2.
Open Basket Type.

Fig. 3.
Flower Basket Type.

Fig. 4.
Classical Roman Type.

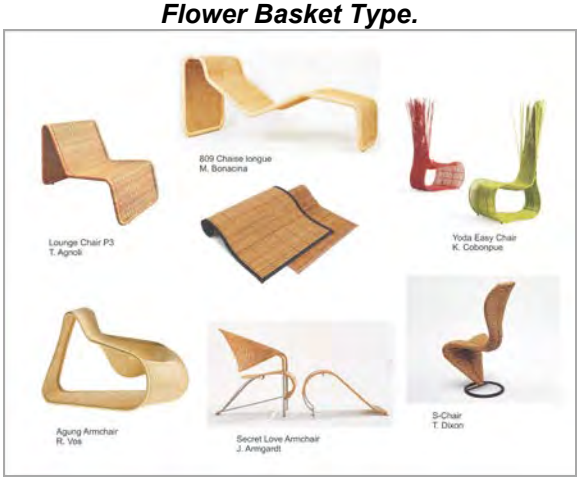

Fig. 5.
Flying Carpet Type.

Fig. 6.
The Tubular Type.

WOVEN FURNITURE: CASE STUDIES

The Case Studies performed for this paper are summarized in the following 5 tables. The findings are systemized and organized following the above archetypal images; the weave is followed where possible. A consistent interest towards wicker was established in the period of the 1950-s and 1990-s as well as the first decade of the 21st century. A clearly defined diversification of form-building, of texture experiments of new weaves and materials is established in the last decade, which tendency can be visually followed in the summarizing tables.

Table 3

Types of rattan chairs according to morphology of design: Open Basket and Tubular type

	Open Flower-Basket Type	Open Basket Type	Tubular Type	Tubular Type
Picture				
Product Name, Materials	Primavera Armchair Rattan	Hanging Egg Chair Wicker or Polycore in several colors	"Su su su" Chair Cane, wicker, rattan, with steel base	Wicker Chair (Idée Lounge Chair) Aluminum, steel, wicker
Year of Design	1967	1959	1966	1990
Author	Franco Albini and Franca Helg	Nanna & Jorgen Ditzel	Kazuhiko Tomita	Marc Newson
Manufacturer	Bonacina 1989, Italy	Sika-Design, Denmark	Bonacina 1989, Italy	Idée, Australia
Morphology of Design, Stylistic Features, Type of Weave, Construction and Details, Material or Material Mix, Analogues	The Primavera Armchair by Franco Albini and Franca Helg, looks exactly like a flower basket. Its' nearest analogue is the E10 Rattan Chair by Egon Eiermann from 1949. The chair is definitely a highly decorative one, reminiscent of the Peacock Chair by Wegner and to other craft examples, because of the fan-shaped backrest. The Primavera's beautiful, airy look is achieved by its openwork diagonal structural mesh, resembling Japanese Ikebana Baskets. Structurally, the weft is being carried by two large rattan hoops, the crossing points are fixed by nails.	The Egg chair by Nanna & Jorgen Ditzel, 1959, follows the typology of hung chairs, such as the Bubble Chair by Eero Aarnio (1968); but this one has a woven surface. It is just one of the many rattan designs by the Danish designer. Morphologically, it is an 'open basket' plus one upholstered seat cushion. The sitter is enclosed, therefore reminding of seaside "Korb Sessel", keeping off the wind and sun. The model is available with structure for hanging; it could hang from the ceiling as well. An analogue is the Eureka Hanging Chair by Giovanni Travasa (from 1958) for Bonacina 1889, where the author exploits a structural mesh for building the rattan surface.	This chair's structure is of the open tubular type, ending in three large openings, containing thicker contour hoops of rattan. This chair reminds us of a piece of garment, since it has two 'sleeves' serving as armrests, although it seems the author meant to represent a smiling complexion. The 'tube' structure gives a soft, all-woven look, the weave is tight. Only the legs remain visible metal. The unusual thing about it is that all three tubular elements are open to the front. The tubular theme brings the visual idea of a springy surface, soft and receding under the weight of the sitter.	The structure is a metal one, woven with rattan on both sides. This is a one-piece organic sculptural furniture item (if we do not count the invisible metal leg behind), with a tight weave. The overall shape reminds one vaguely of a human torso, securing a great angle of the back for a comfortable rest. Itisnoteworthythattheoriginal Feltchair (since the Wicker chair is a version) turnedouttobefitfortherattanweave; thisonlyconfirmstheversatilityofthe material. The structure meets the ground in three points, thus leaving the impression of a free-flowing object in space.

DISCUSSION

The case studies have revealed a wide range of opportunities for designers wishing to use rattan both as a structural material or surface weave. Form-building potential was disclosed to evolve according to the 'basket' type, both open and closed, which brings us to any bulbous shape or its varieties. Next, mixed principles brings metal/wood/rattan structure with caned surfaces, leaving us with all possible surface variations, the oldest of which we called "Classic Roman Type", consisting of an outside covering surface and a seat. Important versions are 'the tubular' type, the bird's nest type, the 'flying carpet' type, etc. Finally, we come to the "structural mesh type" which lacks surface weaving altogether, and leaves us with the structural skeleton woven into a large mesh. De-constructivist approach brings us to the "unfinished" type, which brings pieces closer to natural-looking objects, without, of course, being able to beat the look of the 'bird's nest' type, which is clearly the best.

We can point out the following advantages: purely formally, the pieces are archetypal (the basket image being very strong), bringing characteristic texture and colour of the materials used (rattan, tubular knit, polyethylene, etc.), bringing 'natural' flavour and 'crafty', 'handmade' aroma to such an extent, that the pieces look exclusive. The disadvantage (especially having in mind the exclusivity) is clear: a very high price. Most of the pieces from our case study are exclusive both because they are 'signed' by famous designers, by long hours of expert handwork and the natural material, by the label of the manufacturer, by their exoticism. Being very aware of the price policy of different parts of the world, yet we should acknowledge that this ancient basket-weaving skill brings about some of the best examples of design for garden purposes, public use and for the home.

Table 4

Types of rattan chairs according to morphology of design: "Roman Type" and its versions

	Classical Roman Type	Classic Roman Type	Classic Roman Type	Classic Roman Type
Picture				
Product Name, Materials	Poplar Chair Paper-wrapped high tensile wire	Canasta Series Aluminum frames, interlacing: white or bronze polyethylene	Foglia Chair Metal structure, rattan-core weaving	Ami Chair Stainless steel, expanded PU, handwoven chain tubular knit
Year of Design	1922	2007	1968	2008
Author	Lusty Lloyd-loom design	Patricia Urquiola	Giovanni Travasa	Francesco Rota
Manufacturer	Lusty Lloyd-loom, Great Britain	B&B Italia, Italy	Vittorio Bonacina, Italy	Paola Lenti, Italy
Morphology of Design, Stylistic Features, Type of Weave, Construction and Details, Material or Material Mix, Analogues	The shape of this chair is reminiscent of the ancient Roman woven chairs as a type: it includes a round back, a horse-shoe-shaped seat, the weave surfacing on all sides and under the seat to form a 'skirt'. This chair shape gives rise to many stylized and simplified versions reduced to a curved structural surface plus one seat, in modern design.	"Patricia Urquiola approached the outdoors starting from the theme of woven patterns-reviving and personalizing the concept with a traditional look in mind but giving it a decisively contemporary look without using too much nostalgic influence Vienna straw." The chair was inspired byAsianbaskets (bebitalia.com). The structure is metallic, the weave is "The Vienna Cane", but over-exaggerated by the use of polyethylene strip. In the different pieces, Urquiola follows different types: the "Roman" type for the garden armchairs, the spherical type for the outdoor sofa.	The chair has two parts: seat plus sculptural curved 'kimono' part with two short front legs. Contour and legs are in metal. The weave is structural and tight. The high back brings a different character of the chair, although structurally it belongs to the "Roman Type", it has the look of a fan-shaped high-backed traditional craft chair, giving the sitter 'protection' above his head.	Fully in the vogue of using tubular knit, this piece makes good use of the structural weave of the company's "Chain"knit tubular material. To soften the bumps, cushions are provided. This example proves that the woven pieces vary widely in materials. The knit material adds color and texture over the metal structure.

Table 5

Types of rattan chairs according to morphology of design: "Flying Carpet Type" and its versions

	3D Surface Following Structure	Flying Carpet Type	Flying Carpet Type	Flying Carpet Type
Picture				
Product Name, Materials	Manta Chair The frame is made from metal and rattan	809 Chaise longue Internal steel frame, woven rattan core	Lounge Chair, Model P3 Tubular lacquered steel frame, woven wicker	S Chair Metal structure, cord or rattan; other materials
Year of Design	1998	Ca. 1966	1960 to 1969	1991
Author	D'Urbino and Lomazzi	Mario Bonacina	Tito Agnoli	Tom Dixon
Manufacturer	Bonacina 1889, Italy	Bonacina 1889, Italy	Pierantonio Bonacina, Italy	Capellini, Italy
Morphology of Design, Stylistic Features, Type of Weave, Construction and Details, Material or Material Mix, Analogues	'3D CURVED surface'. Curved surfacing in one single part, metal structural parts: two legs and one backrest contour, covered in a tight polyethylene cord weave. A very important feature of the woven surface is exploited: the ability to cover all complex surfaces.	This Chaise Longue by Mario Bonacina is an obvious analogue of Tito Agnoli's Lounge Chair, with certain variations. The form building principle of using a basic metal structural frame for the sitting/lying support, covered with thick rattan weave, is clearly followed.	This model by Tito Agnoli exploits an idea of seating furniture as a single frame bent in space to provide backrest and seat, that originated in Bauhaus cantilever chairs. Typical for the 60-s and 70-s, this form gave many versions with more or less complex surface.	This chair has a metal structure, over which cord or rattan or other materials are used. The paper cord version is stuffed with grass. The chair has a one-piece morphology, with a metal circular base. The individual feature is the stuffing with straw inside the woven double walls of the chair. An iconic and minimalistic image.

Table 6

Types of rattan chairs according to Characteristic Weaves

	Unfinished Weave	African Basket Weave	Closed Shape, Open Weave	Openwork Mesh Type
Picture				
Product Name, Materials	Yoda Easy Chair Rattan, nylon, steel	Afra Chair Stainless steel structure, hand-woven upholstery with cord and Aquatech yarn	Balou, Loveseat and Sofa abaca, rattan, nylon, steel	Nautica, hanging chair Structure in peeled and tinted natural rattan 32 mm diameter
Year of Design	2008	2016	2014	2014
Author	Kenneth Cobonpue	Francesco Rota	Kenneth Cobonpue	Alberto Sánchez (Mut Design)
Manufacturer	Kenneth Cobonpue, Philippines	Paola Lenti, Italy	Kenneth Cobonpue, Philippines	EXPORMIM, Spain
Morphology of Design, Stylistic Features, Type of Weave, Construction and Details, Material or Material Mix, Analogues	The Yoda Easy chair has the 'unfinished' effect: it reminds us of deconstructed structural textile, where only the warp remains sticking out. The Yoda Easy Chair achieves a tall silhouette, thus giving a hierarchical meaning, a throne-like structure, that: a. acts really decorative; b. makes a screen-like performance, when we use several chairs together, thus separating the space in front from the rest of the room. The weave is open, bringing a basket quality to this piece.	The clean and minimalistic form of this armchair is totally dictated by the technique, used by African women to weave their baskets. This is in fact the 'coiled basket' technique where a thick cord or straw or seagrass is woven and sewn with yarn. The monolithic quality of the armchair does not distract the attention from the exotic weave.	In spite of the large size, this piece has a unique light quality due to the open weave and the fact that we can see through it. The piece is light and transparent, and is manufactured in two versions: indoor and outdoor, with different materials. All models by Cobonpue have the unmistakable visual quality of the different weaves used.	The open weave of this rattan chair relies totally on the decorative effect of the sinuous rattan poles. The swaying movement of the hanging chair finds its visual effect in the wavy line of the rattan poles. Although no weaving is present, the transparent poles also carry a 'trace' of knitted yarn in its meandering forms.

Table 7

Types of rattan chairs according to morphology of design: "Closed Spherical Type" and its versions

	Closed Spherical Type	Closed 3D Shape	Closed Spherical Type	A Compound Configuration of Several Closed Volumes
Picture				
Product Name, Materials	Palla Chair Rattan	Sushi Daybed Water-hyacinth (Eichhorniacrassipes)	The Spaghetti set Natural liana, rattan	Doeloe lounge chair
Year of Design	1960	2008		2010
Author	Giovanni Travasa	Bannavis Andrew Sribyatta	Bannavis Andrew Sribyatta	Abie Abdillah
Manufacturer	BONACINA 1889, Italy	PIE Studio Furniture (Project Import Export)	PIE Studio Furniture (Project Import Export)	
Morphology of Design, Stylistic Features, Type of Weave, Construction and Details, Material or Material Mix, Analogues	In Italian 'palla' means "ball". This type of closed containers resembles spherical shapes, although the sphere is distorted to receive a seat. The weave is really tight and structural, although it is very probable that the structure incorporates metal skeleton. Sculptural effect, many analogues, mostly shaped as an ovoid and serving as poufs, and also made of different materials (e.g. tubular knit).	Here, the type is a closed one with a structural tight weave, the material used is water-hyacinth (*Eichhorniacrassipes*), an aquatic floating plant known to cause great problems by being an invasive species outside its native range. The simple spherical volume acquires a more complex three-pointed topography, which receives the sitter in its recess.	This one, although clearly 'spherical' in type, has an overpowering 'knit', which could also be dubbed 'bird's nest', giving the natural and exotic look of this product. Analogue is "Noodles" by Kenneth Cobonpue.	Structural mesh type. "The design of 'Oplet' from the 30s to the late 70s, a small car used in Jakarta for public transportation, was the starting point and form reference of 'Doeloe lounge chair'. The designer used curved rattan poles as a frame and backrest for the upholstered seating unit. Viewed from the front, the arm rests resemble the circular headlights of the traditional vehicle" (designboom.com) The weave consists of a structural mesh of rattan poles. An analogue is the Eureka Hanging Chair by Giovanni Travasa.

CONCLUSION

Apart from the obvious variety of shape, texture and material, we are of the opinion that there are still unexplored areas of craftsmanship bringing more sculptural potential into the hands of modern design, with a strong ethnic flavour. Woven furniture remains partly on the verge of two separate economic zones: the one of the mass-produced serial objects and the one of exclusive handwork mastery. As usual the contacts achieved between the two bring unexpected creativity of design works, of which we expect to see more in future.

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PRINCIPLES OF DESIGN AND CONSTRUCTION OF FURNITURE FOR THE ELDERLY

Vasiliki KAMPERIDOU
Aristotle University of Thessaloniki, Faculty of Forestry and Natural Environment, Laboratory of Wood Products and Furniture Technology, 54124, Thessaloniki, Greece, Tel: 0030 2310 631046, Fax: 0030 2310 998947, E-mail: vkamperi@for.auth.gr

Abstract

In this study an attempt to approach and record the requirements and needs of elderly people using furniture in their everyday life in the years of economical crisis is implemented. The study used around 100 interviews with elderly people that were carried out on a continual process that lasted around 1.5 years in several furniture stores of Thessaloniki (North Greece), gathering data and information in a frame of conversations, trying to reach a deeper understanding of their habits, needs and requirements related to furniture. The results, were also correlated to corresponding literature, and revealed common wishes and needs for furniture that provide comfort, safety, functionality, pleasure and independence, since furniture could help elderly people continue to be active and self autonomous.

Key words: *design; elderly; ergonomics; furniture; stability.*

INTRODUCTION

Low birth rates and higher life expectancy increase the average age of Europe. Aged people (65 years or over) will likely account for an increasing share of the total population, for 28.7 % of the EU population by 2080, compared to 18.2 % in 2013 (Eurostat, 2013). Within the EE, the proportion of people aged 65 and over in the total population increased by 3.7% during the analysis period to reach 17.4%.

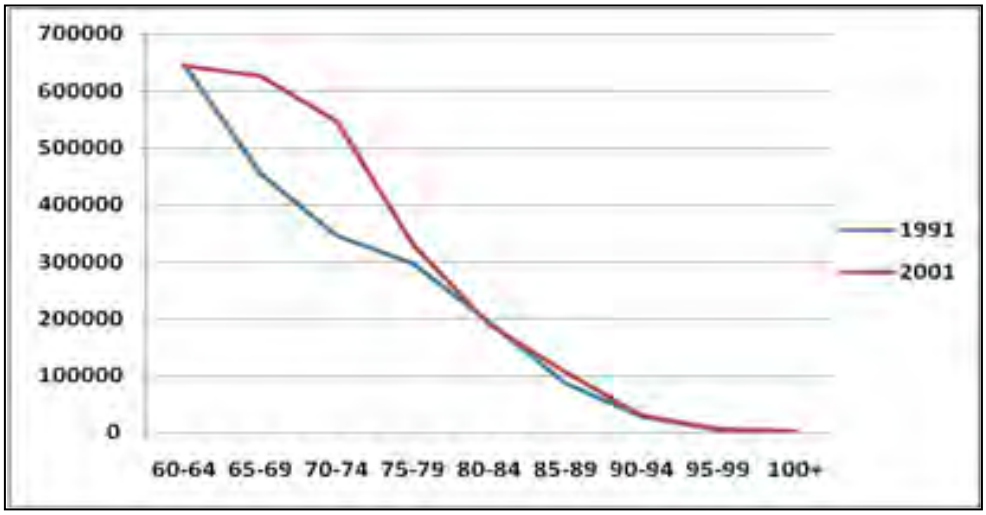

Fig. 1.
Population of aged people in Greece in the years 1991 and 2001.

Similarly, in Greece the average age has increased rapidly in recent decades starting from 28.9 in 1965 to 41.7 in 2010 (Eurostat 2013) (Fig.1). As the number of senior citizens increases, the number of people with disabilities also increases (Hrovatin 2012). This fact imposes the obligation on furniture designers to search for solutions that would at the same time combine elegance, comfort and safety requirements. These complex design methods that create furniture products physically and psychologically adapted to the user's needs base on ergonomics (Klos et al. 2014). People of third age live longer, are healthy and active longer and have different expectations on living conditions, than in any previous age (Jonsson 2013).

Even though the economic crisis has influenced the shopping behavior of all people, elderly between 65 and 69 still consume and spend more money on furniture than younger people. Despite of

this, furniture manufacturers seem to lack knowledge about the diverse needs of aged people, referring to furniture, mainly because of a poor communication between furniture industry and its end-users (Jonsson 2013).

OBJECTIVE

The objective of this study is to increase knowledge and awareness about the ways in which elderly people act on, are influenced by, reflect on and utilize furniture in their home environment the current years and specifically, aim is the communication and bringing of end-user opinion and experiences into sight, in order to improve the conditions of furniture design, intended for old people and their housing environment conditions in home. This human centered approach expect to inspire the world of designers and manufacturers to generate new design practices, that will hopefully contribute in developing furniture solutions that suit the specific needs of the elderly and improve their sense of coherence according to their own abilities, skills and experiences, maintaining a sense of dignity and independence.

MATERIAL, METHOD, EQUIPMENT

In this study an attempt to clarify and record the requirements and needs of elderly people using furniture in their everyday life is implemented. A number of around 100 interviews with elderly people was carried out on a continual process that lasted around 1.5 years in several furniture stores of Thessaloniki city (Greece). Data and information were gathered in the frame of conversation about their upcoming shopping process and experience, where their attitude about furniture choices and solutions, their interests, needs, expectations, concerns and reflections related to furniture were discussed, trying to reach a deeper understanding of their requirements, under a relaxed atmosphere without forcing people to complete specific questionnaires. Some of the participants were already aware and had a clear idea of their requirements, needs or problems related to furniture products and it was easy to analyze them, while sometimes elderly people have the problems and needs confusedly on their minds and posing additional questions is necessary to clarify their attitudes. Of course, through this process one could record only the thoughts and expectations that they have at the moment of conversation according to their experiences, but yet they had not realized the possibility of using and enjoying new improved furniture products and solutions, that is the reason why further discussion with several hypothetical questions were carried out. Their attitudes and recorded data were correlated to previous works of literature.

Aged people often do not seem so willing to spend their time, completing questionnaires, unlike developing a conversation, mainly due to their need to communicate and share their experiences. 90% of the participants were between the age of 60 and 70, while only 10% of them were between 70 and 75. An attempt was made to include in the same percentage both genders, singles as well as couples of elderly and people coming from diverse social and economical backgrounds. Processing of the recorded information was implemented and the most significant concerns and requirements of the aged participants that were drawn from this process are summarized hereupon.

RESULTS AND DISCUSSION

According to the results of processing of the data and information coming from the research process of this work and the record of results of previous researches, an attempt was made to categorize the needs, concerns and expectations of aged people, resulting in the following main categories of furniture usability aspects.

Dimensions

A frequent problem seem to be a mismatch between the elderly body dimensions and their furniture. The chairs and beds are often too high or deep and tables are often high for the elderly, presenting a negative effect on the sitting posture of the elderly especially while reading or writing. Chairs and sofas should be chosen according to the size of the person. Taller people require generally deeper seats, while smaller persons need shallower seats. Dining/coffee tables should also be of appropriate height for the user. Cabinets and wardrobes should be chosen after considering accurately the variable storage space needs. Storage requires also low physical effort in using as well as having ergonomic dimensions for the elderly. Important is also the relation between the dimensions of the house and rooms, compared to furniture dimensions, because this affects the interior free space left to the Elderly to move freely and the possibility of maneuvering the furniture units. A possible solution would be the furniture to be in some way height-adjustable. The dimensionally adequacy call

for the attention of furniture designers and manufacturers to take into account the sizes and dimensions of aged people bodies and home interior spaces.

Cleaning and Hygiene

Elderly people are more susceptible to infection and diseases, necessitating greater care to achieve hygienic conditions within the house (Hrovatin 2012). Additionally, they spend a great deal of time indoors, due to social and financial reasons. Therefore, following the basic hygienic standards is crucial to their health condition and particular care needs to be taken in the design of kitchen furniture, where food is prepared, as well as furniture where they prefer to seat, sleep or spend much time, as well as furniture of bathroom. Generally, conversations with elderly people showed that they require of properties that make furniture easier to clean, such as flat surfaces, non-porous, resistant materials to scratches, smooth and without joints or seams, simplicity of design in order to have access during the cleaning process to the whole furniture and the opportunity for the user to clean the floor beneath the furniture and reach there with a vacuum cleaner easily. The upholstery should provide the capability to be cleaned easily and to be frequently removable and washable and all the upholstery materials should be impenetrable to prevent surface soiling. Waterproof seat cover materials could be used for the protection of the seat cushion from soiling and make the seat easily cleanable but these materials normally have poor breathability properties, which makes them uncomfortable to sit on and increases the risk of pressure sores (Meinander and Varheenmaa 2002).

Transfer

Furniture is required to be easy to move and not too big and bulky. They should be easy to get in and out of the room and probably the house. Also, they could be movable with the help of wheels, which is an easier way and do not damage the floors (Jonsson 2013). Materials such as solid wood or lightweight honeycomb panels may be used in furniture construction, in order to ensure easy transfer.

Access

Old people appreciate products that are accessible from many diverse users. Easily maneuverable levers and controls facilitate use by people with reduced tactile sense or no sense of touch in hand. The placement of furniture and parts of it must not challenge or frustrate old people suffering from reduced ability to stretch and reach (Jonsson 2013). Additionally, as everyone requires enough storage space, as well as elderly people, this storage should be easily accessible. For instance, under the bed storage might not be a good idea because it involves bending, or manipulating the bed. Dressers with drawers that glide out easily could be a good choice.

Stability and Safety (preventing falls and accidents)

For old people that have slower reactions and grasp reflexes, sharp edges and corners of furniture may pose great risks. Furniture should be stable, offer grip supports and be difficult to overturn in order to reduce the risk of falling. Potential hazards could be inadequate lighting, loose rugs, unstable furniture. It is dangerous to use doors with unmarked glass, handles not identifiable, and furniture that are the same color as the wall or have sharp corners, jutting bases etc. (Pinto et al 1997). Necessary seem to be for elderly people the existence of sliding doors with long horizontal handles, kitchen base cabinets on easy-to-roll castors and an appropriate way of placement of the furniture in house. Research has shown that adapting living space to the needs of the elderly could reduce the risk of injury by 30% - 50% (Hrovatin 2012).

In kitchen there should be provisions of continuous knee space beneath the countertop, the cooktop, and the sink. Due to balance disturbances of the elderly there is the necessity of rounding the worktops, replacing traditional doors with a vertical axis of rotation with sliding doors or doors opening in the vertical plane, as well as using recessed handles or push-opener mechanisms. The cabinet width of 600 or 1000mm were proposed as ideal in previous researches, with sides at the angle of 85º. The fronts could be equipped with handle strips. Cabinet lightning could be fitted with a movement detector. The cabinets could have fittings elevating the front in vertical position (Klos et al. 2014). Shelves and appliances should be in right height, worktops should be regularly arranged, cleaning inside the corners or shelves under the cupboards should be easy. A carefully planned arrangement of kitchen elements ensures the safe working with the minimum required motion (Hrovatin 2012).

Seats are required to be firm in order to help the elderly not to sink into the furniture making it difficult to get up. Chair armrests should be sturdy, in order to help the elderly to get out of the seat and strong enough to handle the weight of the person, because elderly are not always strong enough to lift their bodies. Suitable for aged people could be also chairs with electric mechanisms that look similar to a recliner but in fact tilt forward, helping them get out and stand. Upholstery should not let

the body slip down the sofa and fabric upholstery is better to be preferred instead of polished leather or vinyl. The bottom of the legs of furniture should be non-slip, so that the item does not move when the elderly are trying to get out of it. High tables and chairs seem to be better as people can get in and out of them easily. Round tables or tables with rounded edges are preferred by them because they minimize injuries. Shelves or bookcases should be secured to the wall or fastened with safety straps to prevent tip over. Shelves should not be overburdened.

Signage with big clear icons appeared to be important for aged people, since it helps them recognize the different surfaces, warns them about edges, corners, dangers (Leung et al., 2012). Seats should be of a color that contrasts to the surrounding area, also should be in the range of 450mm to 475mm high and a recommended width of 500mm with firmly padded seats incorporating rounded front edges.

Flexibility

Elderly seem to require a large range of adjustable tables and chairs, because there are several body sizes and movements. Lift chairs are an option for anyone who has difficulty getting in and out of a seated position. Chairs should generally allow changing sitting positions in an easy way, because constrained sitting is bad for health, contributes to chronic disorders, muscle pain, impaired circulation (Jonsson 2013). Beds should allow raising and lowering of each end separately and appropriate mattress that provides needed support. Products should be smart, easy to use and not complicated, since aging people do not become more flexible with time.

Health - Ergonomics

Ergonomics optimizes performance and productivity, reducing the risks of injury, discomfort and disease (Pinto et al. 1997). When materials and mattresses conform to specific anthropometrical and physiological-hygienic conditions, furniture can satisfy human needs in rest and sleep and help the body to recover. The shape, color, texture and their constructional parts play an important role in furniture use. Supine position contributes in releasing of the body weight and to a more intensive resting phase. A complete and maximal relaxation of muscles occurs only when optimal matching of the form of the upholstered furniture and the shape of the user's figure is possible allowing the natural shape and course of the line of the spinal column. Beds for example should correspond to dimensions of user, ensure proper alignment of the body during sleep (shape of the spine column and other body parts), ensure even load of the body upon the lying surface, ensure air-permeability, thermal conductivity, moisture permeability, correspond to different body weights and meet hygienic/health standards (Smardzewski et al. 2005).

The ease of ingress and egress of seating furniture depends on its dimensions, the position of the armrests, whether the user is able to put the feet in the space underneath the seat pan and the angle of the backrest. Head and neck rests promote an experience of comfort. Soft seat pans should be thick enough for the user not to feel the hard surface underneath it and the compression of the material should be similar to that of human tissue, even make recommendations regarding optimal load distribution for different regions of the body. The need for warmth and cooling increases because the ability of old people to regulate their body temperatures changes. Inflexible sitting positions constitute risk factors causing neck problems, shoulder problems and back problems (Jonsson 2013). Recliners can be useful for seniors. They are often used for sleeping at night because of medical conditions that cause breathing difficulties or when legs need to be elevated for better circulation. Footstool sometimes are necessary for rest of legs and also allows changing one's sitting position. Some furniture enable the performance of specific activities in addition to their appliances usages, for example support to holds up a book when reading, transporting objects or sitting during other activities (Jonsson 2013). Steel chairs are too cold and hard for the elderly (Leung et al. 2012). Pillows, for beds, chairs or sofas can add comfort, as the user can arrange them for additional support. Elderly people with highly sensitive skin should avoid clothing with hard seams and sharp wrinkles and choose materials that feel pleasant. Upholstery should provide thermal comfort and transmission of moisture from the skin, also have good mechanical durability (surface smoothness, protruding coarse fibres, friction, elasticity, abrasion and tear resistance) and should not generate allergic reactions. Wool, raw silk, rubber and monomers containing polyamide should be avoided (Meinander and Varheenmaa 2002).

Durability and Maintainability

Furniture should withstand wear and tear, cleaning and washing, demand a minimum of cleaning, such as surfaces that hide dust (Jonsson 2013). Wood was mentioned as a preferred material, because it is believed to be a strong, durable material that is associated with quality and high

aesthetics. Furniture should be stable and durable, with high elasticity, with strong and tight joints and assembled with fittings or adhesives of high quality. Among others, elderly face problems with disassembled joints in chairs, sofas and low quality of hinges in cabinets.

Approaching the identity – Attractive appearance

As it was revealed, furniture can be significant for the elderly because reflect a sense of home and inspire a familiarity. The elderly require coherence between existing furniture in their homes and the new. Furniture could reflect independence, relaxation, safety, joy, could be modern, timeless and people in the design process could be closely involved in creation of knowledge on elderly people's attitudes giving feedback to the design (Jonsson 2013). They prefer furniture to have style that has a relaxing impact on the body and mind that helps people calm down, alleviate the symptoms of stress. They prefer warm pastel colors that increase optimism and influence positively the activity of human body (Fabisiak and Hrovatin 2014).

CONCLUSIONS

The results showed that furniture of aged people should be comfortable, ergonomic, practical, durable, easy to clean, of suitable dimensions, harmonious and should suit the individual, environment, usage and the human body, in order to support and enrich old people for as long period of their lives as possible. They need their homes to reflect their own identities and personalities, to promote independence and also to contribute towards creating the greatest feeling of dignity, sense-making and freedom possible, which is of great significance to quality of life and well-being. In order for this to be possible, designers should be closely involved in the process of generating knowledge concerning the needs of old people, which ought to extend to attitudes that may be inherent in the design of furniture and a holistic view on humans and their diverse needs and expectations.

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GUIDE FOR VALUE-ADDING CONTEMPORARY ARCHITECTURE AND FURNITURE DESIGN IN VERNACULAR SPIRIT

Biborka BARTHA
Arch. PhD – TRANSILVANIA University in Brasov – Faculty of Wood Engineering
Address: B-dul Eroilor nr. 29, 50036 Brasov, Romania
E-mail: bartha_biborka@yahoo.com

Alin M. OLĂRESCU
Eng. PhD – TRANSILVANIA University in Brasov – Faculty of Wood Engineering
Address: B-dul Eroilor nr. 29, 50036 Brasov, Romania
E-mail: a.olarescu@unitbv.ro

Abstract

The purpose of this study is to embrace regionalism and specific Romanian wood construction methods and create a vital connection between vernacular concepts and new interior design. The methods used in the research are based on field trips, visiting Romanian villages, connecting with the community and determining the degree of presence of traditional wood structures and housing, the transition of vernacular construction typologies in the contemporary context.

The aim of the project is to create furniture with value and meaning which increases with time and through the use of local communities. This type of interior design achieves meaning as a part of the variety of cultures of the local Romanian communities and as a symbolic reflection of the context. The objective of the experimental investigations was to determine specific local characteristics of vernacular architecture and furniture in Romania and specific European countries. The results and conclusions of this investigation also focus upon manifestations of neo-vernacular architecture in national and European context and set the basis for the main value-adding methods proposed in this research.

Key words*: vernacular architecture; vernacular furniture; value-adding; rural context; methods; wood.*

INTRODUCTION

The vernacular house embodies a complex set of elements of everyday life - occupation, needs and activities of the inhabitants. The traditional Romanian wooden construction is a result of imagination, but also a direct reflection of the needs and the specific lifestyle of several generations. These functional elements have been in an interdependent relationship with the structural ones, along with the change regarding the needs of residents, new solutions had to be found in order to satisfy the inhabitant's needs on both levels. Thus, we can observe links between the spatial layout evolution of vernacular housing (due to functional reasons and specific needs) and the structural developments that made possible the diversification of wooden joints which led to various architectural, functional, and stylistic forms.

The aim of the experimental investigations was to determine specific local characteristics of vernacular architecture and furniture in Romania and specific European countries. The results and conclusions of this investigation also focus upon manifestations of neo-vernacular architecture in national and European context and set the basis for the main value-adding methods proposed in this research.

The originality of traditional Romanian architecture had been exploited in the 19th century on a monumental scale and will continue to inspire romanian architects like Ion Mincu in the creation of a neo-Romanian architectural current which had a profoundly reinterpreted vernacular architectural vocabulary. He believed that he had found the „*roots of a windfall*" (Curinschi 1981).

Even though the research of vernacular architecture has existed throughout several decades, there hasn't been identified an organised atempt of systematisation of the results concerning this theme, the interpretation and concept transfer of Romanian vernacular architectural elements being present in the last 300 years on a national level with the aim of representing the spirituality of the Romanian population.

Italian rationalist architects of *Gruppo Sette* (Sabatino 2009) stated in 1926 that "*tradition does not disappear, but changes appearance*." This paper starts from this idea that revolutionized and changed the approach of professionals towards the concept of tradition and vernacular. The aim of the research is to present in a synthetic and visual manner forms of architectural-functional-structural-

symbolic evolution of vernacular housing and furniture on a European level in order to obtain strategies of reinterpretation and reintegration of vernacular concepts on a national level. These strategies place vernacular architecture in a new light, making it possible to apprehend traditional architecture as a process of sustainable design with a range of actions on diverse and multiple levels: *the physical* (*natural-* topography, landscape, vegetation, *structural*, *spatial*, *material*-materials, textures) and *the spiritual* (traditions, customs).

Genesis and definition of vernacular concepts

Vernacular architecture represents a set of values on several levels: historical, aesthetic, social and cultural, and is a direct reflection of the user's talent and building techniques. However, in terms of vernacular heritage protection, relevant research and studies are extremely low in number. In this regard, investigations have been made concerning the evolution of the vernacular concept through the activities of several personalities in this field such as: Christopher Alexander, Hassan Fathy, Kenneth Frampton, Paul Oliver, Dell Upton, Bernard Rudofsky and Amos Rapoport.

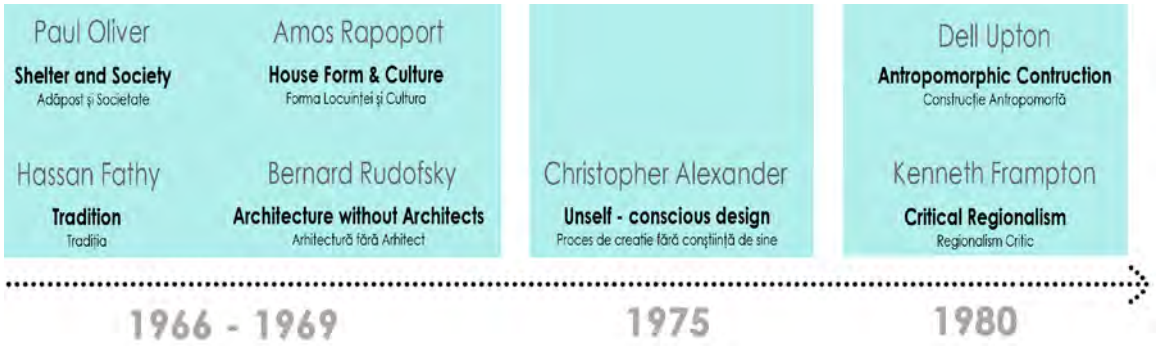

Fig. 1.
The evolution of the vernacular concept.

The role of tradition in the modernization and globalization era has led to a series of debates in the presence of specialists, architects, designers in recent decades (Ruggiero 2009). Tradition is the mass of cultural, religious, ethnographic values and modernity may be associated with cultural transformation, mobility, social class stratification, a consumer-oriented society (Mitrache 2008). At present, tradition and modernity are no longer seen as two opposite concepts, but ones that coexist, overlapping and influencing each other.

Tradition is the first resource for reintegration, rehabilitation and maintenance of local identity, on regional or national level. Kenneth Frampton claimed in the 80s in his book on critical regionalism that local tradition can be a tool for the creation of identity.

Critical Regionalism is an approach in the architectural field that aims to define space and identity by replacing concepts of uniformity and monotony of the International Style, but in the same time rejects individualism and ornamentation of postmodern architecture. The style of critical regionalism corresponds with a type of architecture that is rooted in the tradition of modern architecture, and is linked to the geographical and cultural context. Critical Regionalism may extend its meaning beyond the field of vernacular architecture. It is a progressive approach to the design process that attempts to mediate between global and local manifestations of architecture (Frampton 1983).

OBJECTIVES AND METHOD

The proposed methodology is based on the findings of the synthesis and experimental research. The characteristic vernacular elements were selected and integrated in the context of contemporary process of architecture and furniture design. These features can also be found in the context of European vernacular, such methodology being applicable beyond the local context. This holistic approach consciously seeks to implement value-adding vernacular elements in any context, only small adjustments are necessary or addition regarding the specific place (regardless of culture, population, etc) (Curinschi 1981).

Wood as a primary buiding material is a clear reflection of the local communities way of living. Even though the communication of traditional building methods from one generation to the other has had a fragmented evolution, it has managed to adapt itself to the contemporary context due to the

collective interest of local communities and their way of understanding life, living and the importance of traditional wood contructions.

Wood can be considered one of the first building materials. So far, it has had a constant and varied utilization concerning built environment with or without a structural role. This aspect is mainly the result of the spread of the wooden material as species around the world, but also due to a series of qualities like: easy processing, light weight and good mechanical characteristics. Even if it can be characterized by a high perish ability linked to multiple factors like vulnerability to fire, it still has a wide utilization range in the present.

The first wooden frame structures can be dated back to the year of 200 B.C. (Pănoiu 1977). The first structures were very simple, consisting of branches and / or thin wooden rods connected through wattle work (Lăzărescu 2010) or other joints between larger trunks (Crișan 2003).Regarding Romanian territory, vernacular construction were simple regarding the structural forms, with some of the wooden pillars buried under the earth, the visible part being covered with planks, wattle and clayey soil. These structural systems diversified in time, leading to the construction typology with horizontal logs resting on stone foundations (Olărescu 2012). From a structural viewpoint, these constructions were made of round pillars embedded in the soil, ensuring the stability of the construction (Păcală 1915).

The research results helped develop a methodology that proposes the following steps: consultation of the (new-created) databases, decision making according to the purpose, drawing guidelines, selection of the sustainable option, functional and stylistic interpretation of the elements according to the assumed aims, direct value-adding. This kind of new approach to the design process has to be seen as a contemporary need, a specific need in a globalized world, and aims at giving answers to specialists in search of value-adding elements of local tradition in their architectural and furniture projects.

The applicability of the proposed methodology in the field of architectural and furniture design in harmony with contemporary requirements was checked by a few projects: a sustainable housing unit and an innovative furniture ensemble designed according to the methodological steps that resulted from the research.

Steps for value-adding contemporary architecture and furniture design in vernacular spirit

The proposed methodology is based on the findings of the synthesis and experimental research. The characteristic vernacular elements (see in figs 2 and 3) were selected and integrated in the context of contemporary process of architecture and furniture design. These features can also be found in the context of European vernacular, such methodology being applicable beyond the local context.

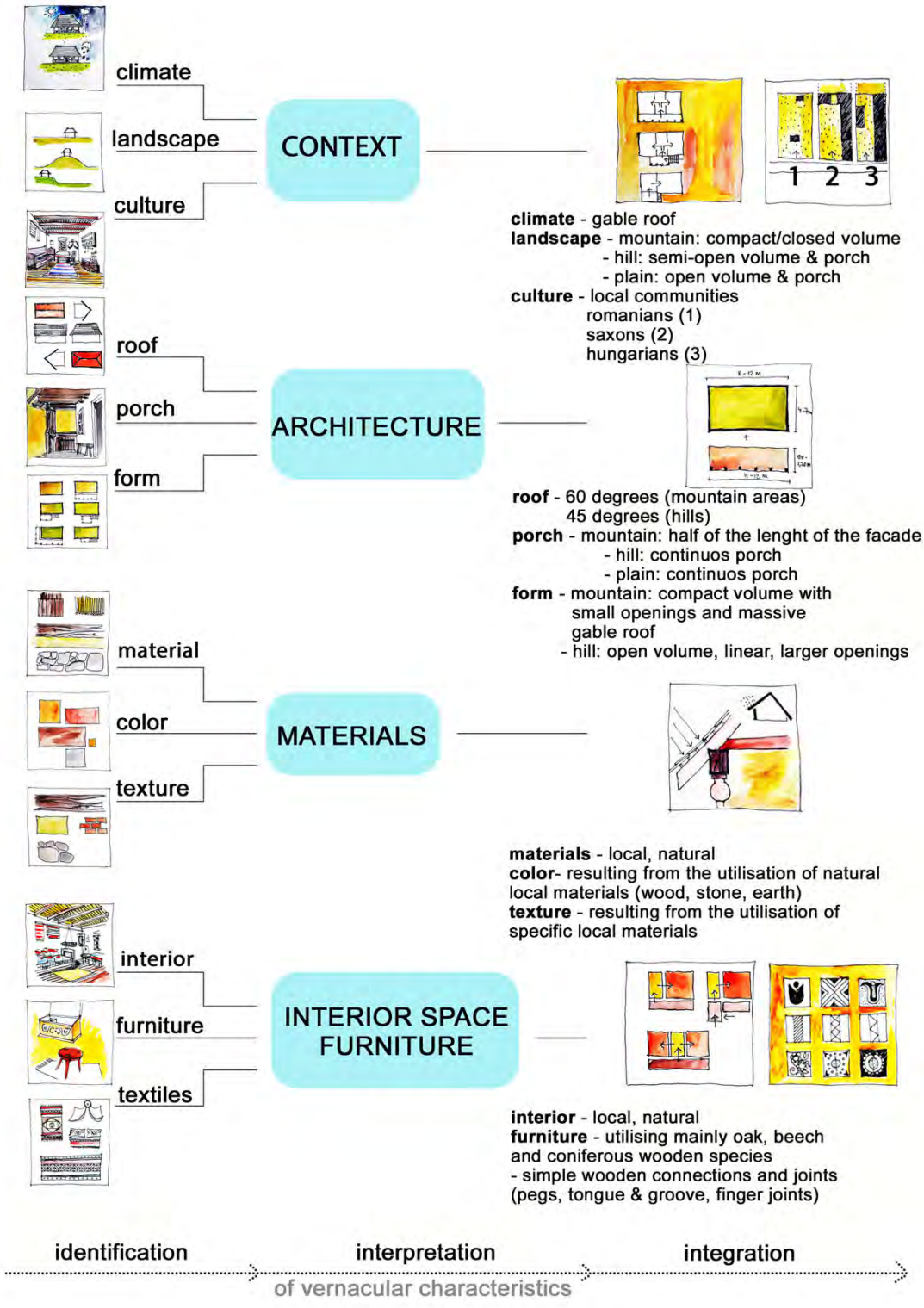

Fig. 2.
Integration of vernacular elements in the process of contemporary architecture.

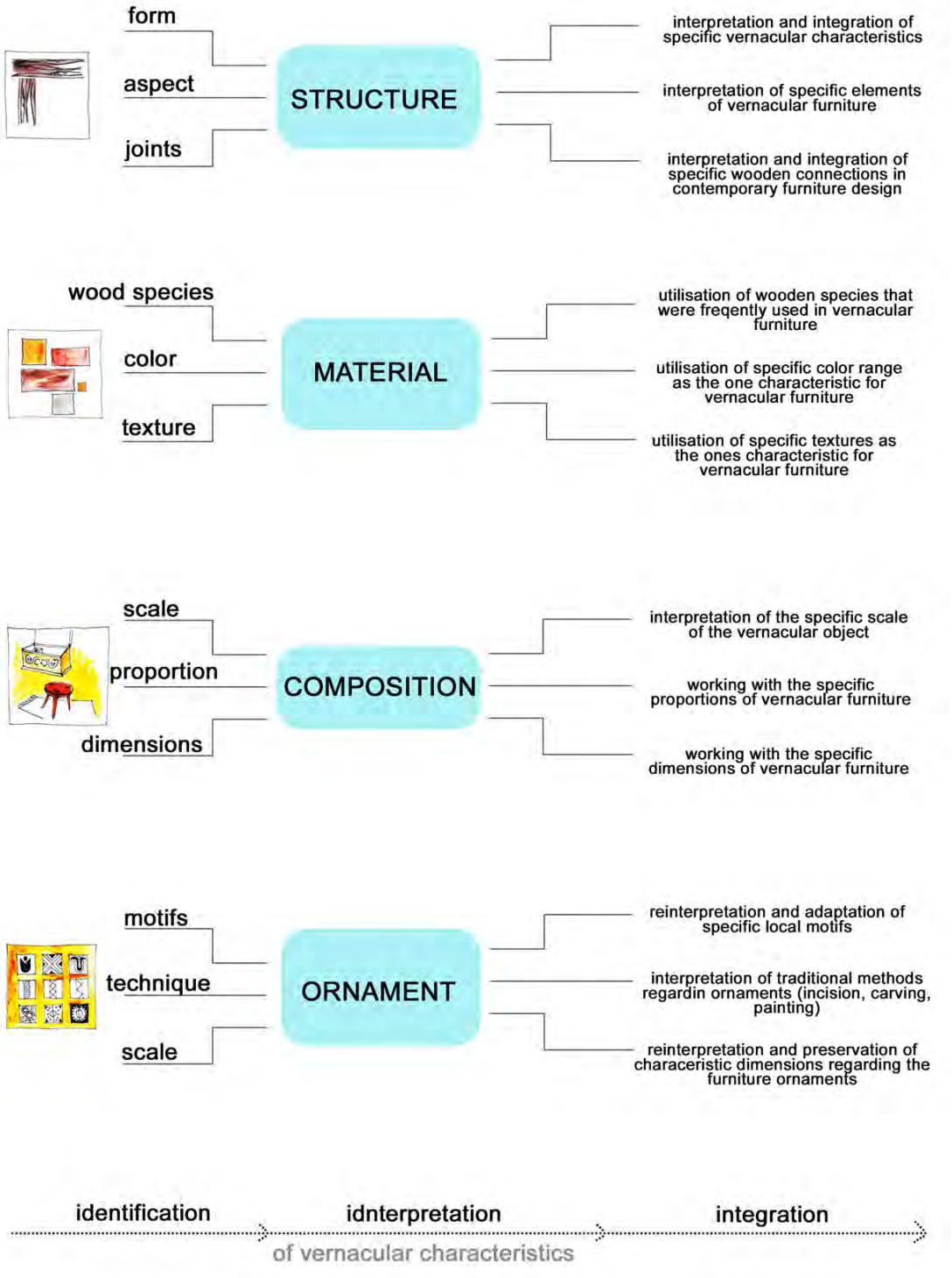

Fig. 3.
Integration of vernacular elements in the process of contemporary furniture.

This holistic approach consciously seeks to implement value-adding vernacular elements in any context, only small adjustments are necessary or addition regarding the specific place (regardless of culture, population, etc).

The stages of the theoretical and experimental research conducted in previous research were based on a holistic approach concerning the following:

- Wood utilization patterns regarding vernacular architecture and furniture;
- Architectural ornaments and furniture;
- Architectural components;
- Manifestations of European vernacular architecture and furniture;
- Manifestations of Romanian vernacular architecture and furniture;
- Effects of the contemporary context upon the vernacular architecture;
- Neo-vernacular architecture and furniture in Europe;
- Neo-vernacular architecture and furniture in Romania.

The methodology is based on the above mentioned research and includes a logical set of steps:

Cycle I - theoretical stages

1) Consultation of existing databases, regarding:
• Current status of vernacular architecture and furniture;
• Specific elements of vernacular architecture and furniture;
• Materials used in vernacular architecture and furniture;
• Volumetric composition, color, facade, solutions of technical and constructive connections between different elements, characteristic joints regarding vernacular furniture;
• Ornamental characteristics depending on the geographical context.

2) Making decisions regarding the final purpose.

3) Selecting the strong/main directions of action:
• existing geographic and climatic context, taking into account a number of the constants on site (typology of rural settlement - the village plan, valley, cultural-, ethnic-, economic-, social context, etc.);
• The components and the operation of the household, needs for comfort;
• Volumetric and specific form of dwelling;
• Construction materials, color, texture;
• Orientation, facades.

Cycle II – practical stages

4) Detection of the sustainable direction:
• Selecting from multiple possible optimal variants/feasible options as required;
• Identification of the optimal measure considering the possibilities and the desired result.

5) Functional and stylistic interpretation of elements
• Assuming and highlighting the characteristic elements of vernacular architecture and furniture on a holistic level: volumetric orientation, textures, materials (in the case of an architecture project); scale, proportion, structure, materials (in the case of furniture design);
• Assuming of detail elements such as specific joints, ornaments characteristic local structural features for walls, roof or concerning furniture items.

6) Direct value-adding through:
• Architectural projects or functional architectural conversion;
• Projects of organization, reorganization of outer space (decorative and functional) and landscape design;
• Projects for furniture and other interior and exterior elements.

A value-adding methodology of vernacular elements on European and national level was realized, comprising six stages grouped into two cycles, first with theoretical stages such as consultation of existing databases, decision making, drawing guidelines; the second cycle comprises the practical steps such as selecting the sustainable direction, functional and stylistic interpretation of the elements according to the aims assumed, direct value-adding through architectural and furniture projects.

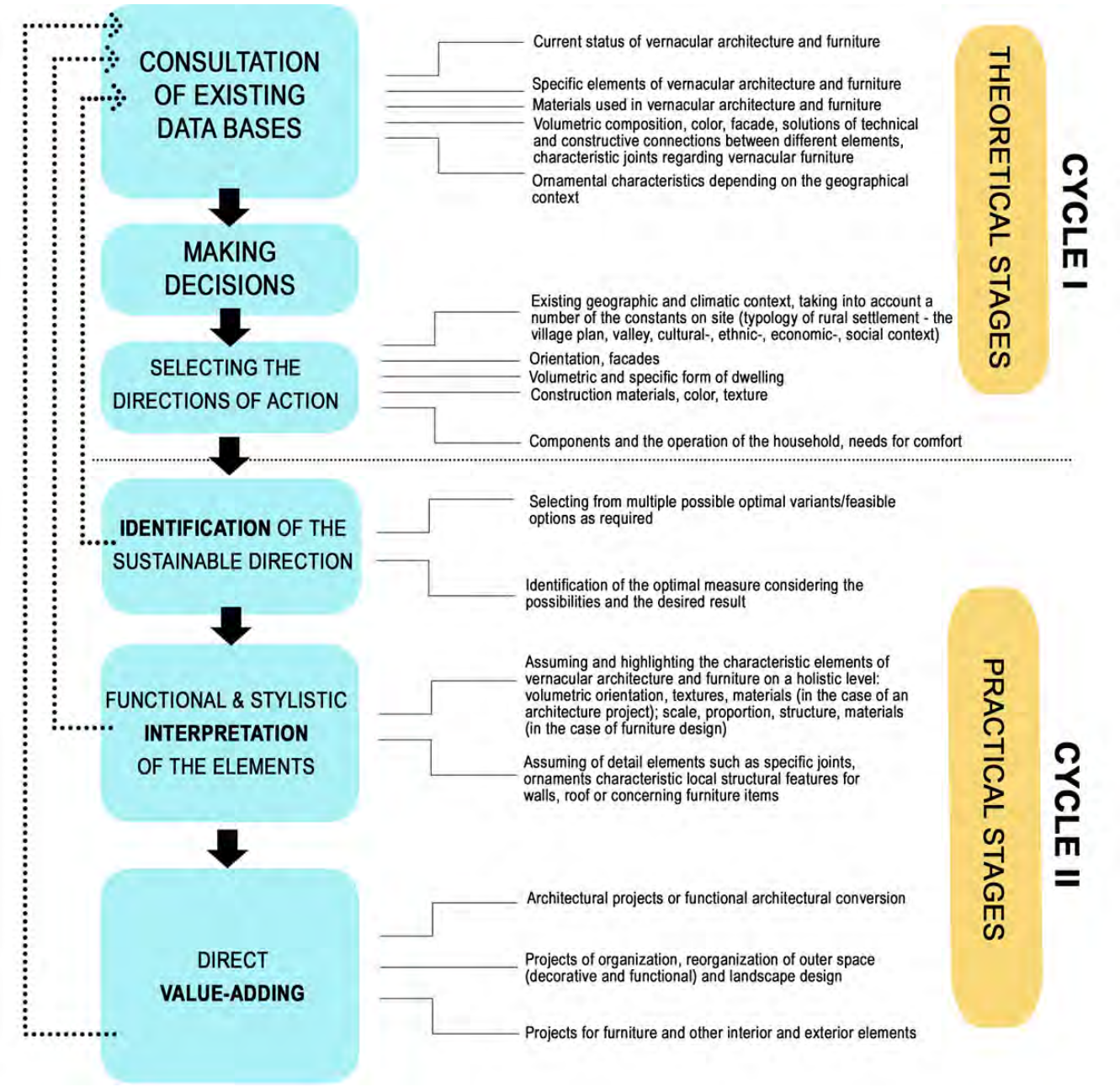

Fig. 4.
Steps of the proposed value-adding methodology.

The figure above contains (Fig. 4.) the steps of the proposed methodology of value-adding vernacular concepts to contemporary design process.

CONCLUSIONS AND DISCUSSIONS

Results

The upcoming figures (5, 6, 7) contain 3 study cases (two regarding contemporary furniture in neo-vernacular spirit and a house project) which exemplify the application of the proposed methodological steps for value-adding contemporary architecture and furniture. The main concepts that are taken into consideration for reinterpretation are the following: structure (form, aspect, and wooden connections/joints); material (wood species, color, and texture); composition (scale, proportion, and dimensions); ornaments (motifs, technique, and scale).

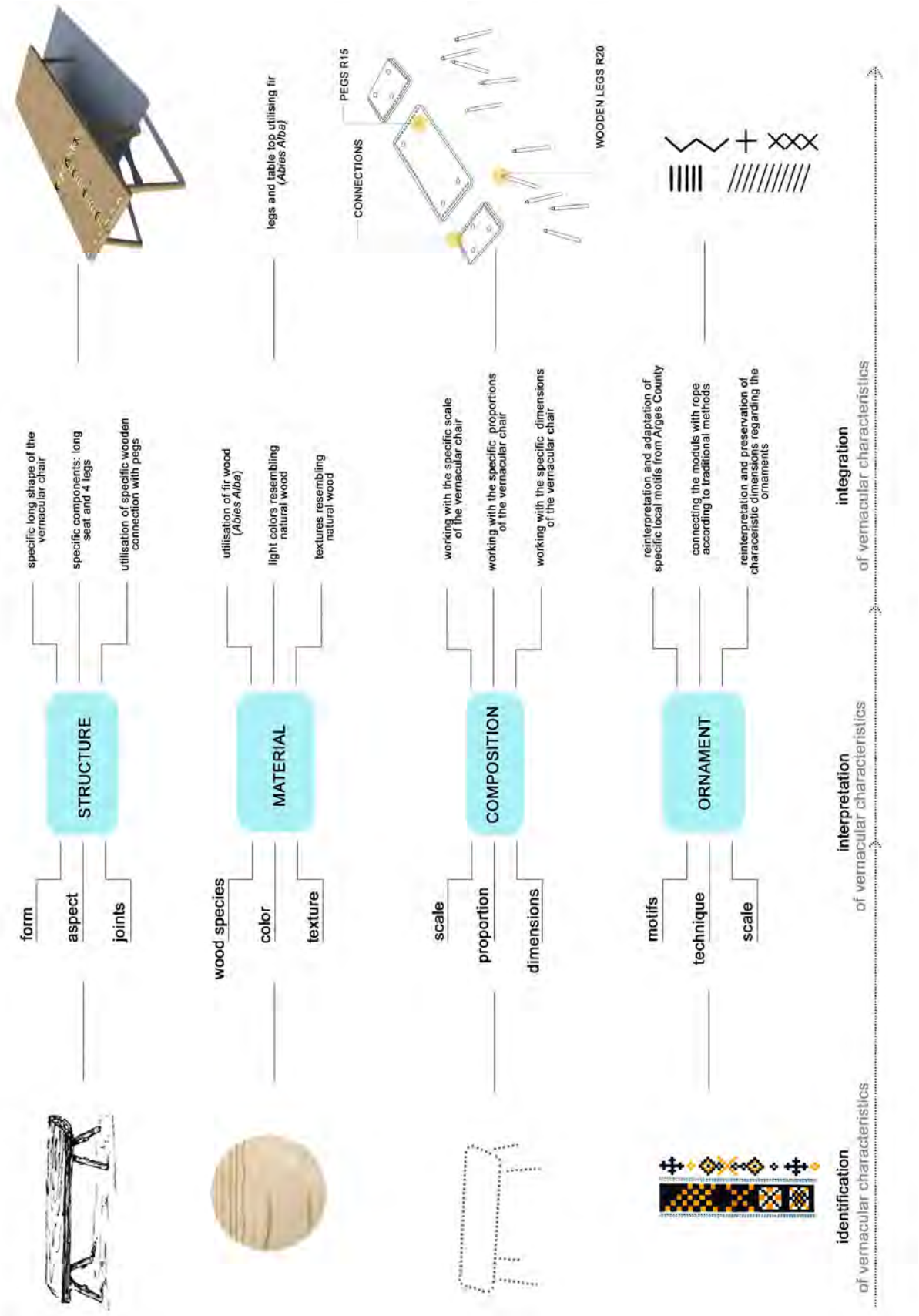

Fig. 5.
Application of the proposed value-adding methodology for contemporary furniture, case study 1.

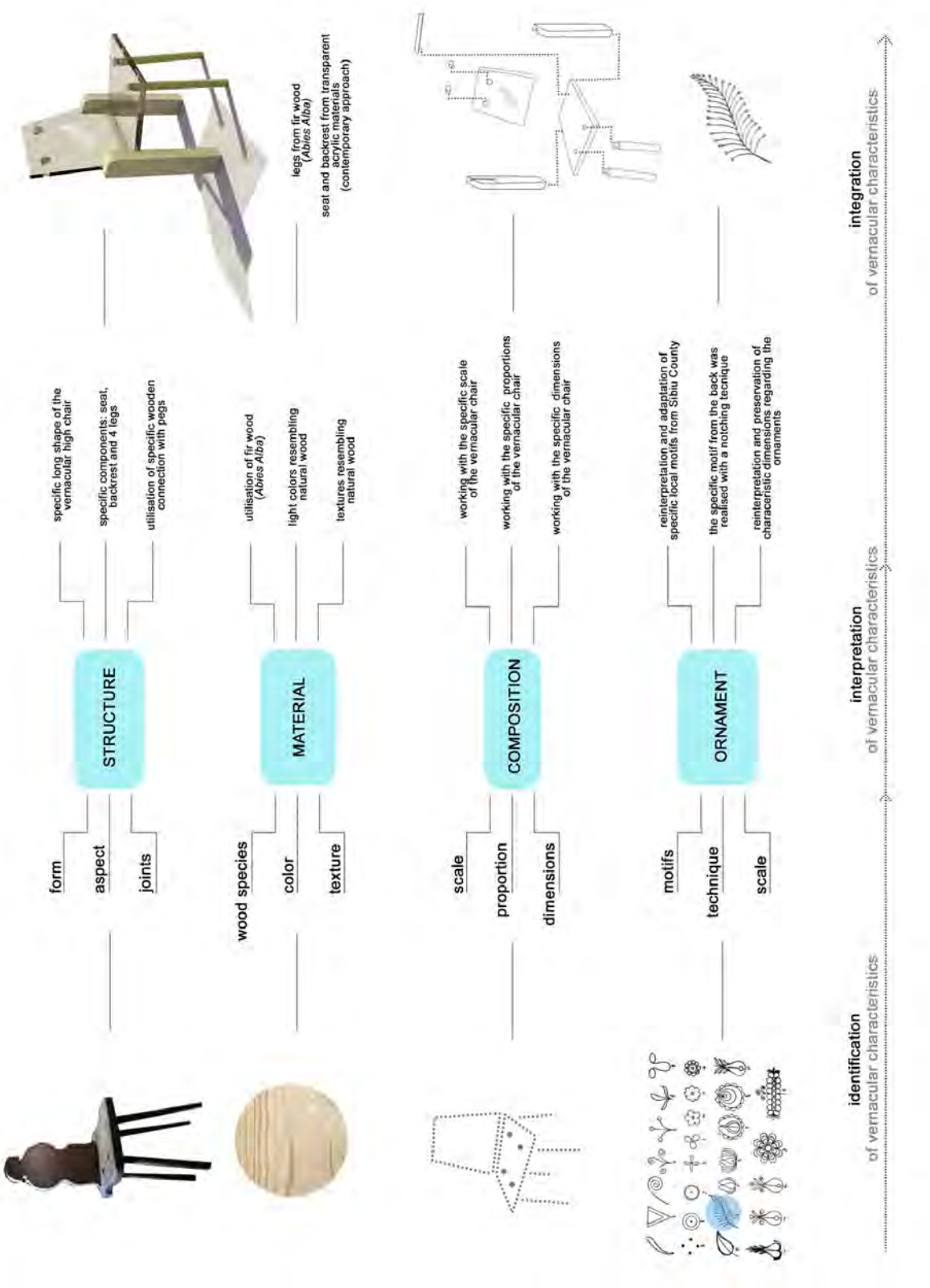

Fig. 6.
Application of the proposed value-adding methodology for contemporary furniture, case study 2.

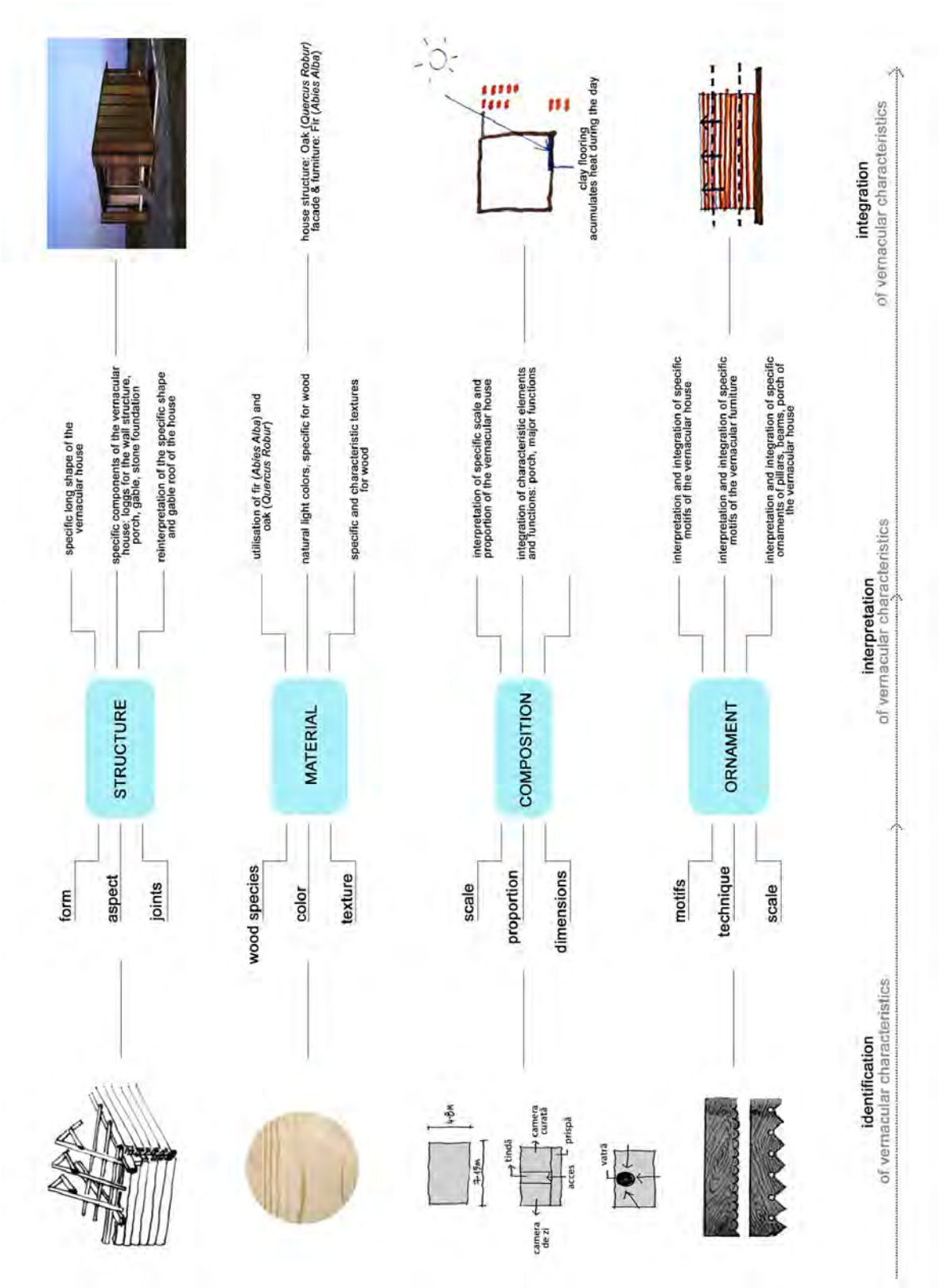

Fig. 7.
Application of the proposed value-adding methodology for contemporary architecture, case study 3.

CONCLUSIONS

The research embraces *regionalism* and *specific Romanian wood construction methods* and defines a clear connection between vernacular concepts and new interior design process (Fig.3)

concerning Romanian villages, connecting with the community and determining the degree of presence of traditional wood structures and housing, the transition of vernacular construction typologies in the contemporary context. The social layer of the study has completed the architectural, aesthetic and functional aspect of the analysis with the purpose of offering a more profound approach in the process of identification of *traditional characteristics of Romanian architecture and wood construction methods* and a guide for integrating them in the present design process. This type of design process achieves meaning as a part of the variety of cultures of the local Romanian communities and as a symbolic reflection of the context.

The differences between the "vernacular" and "modern" design processes are very clear: in the case of vernacular, the production would be singular, crafted and local, whereas the contemporary modern production would be characterized by a serial, industrial, dislocated approach. In order to achieve the wanted result, we need to utilize and rely on the advantages of modern production, but should not forget that the design should reflect the profoundness of a local vernacular concept.

The research results helped develop a methodology that proposes the following steps: consultation of the (new-created) databases, decision making according to the purpose, drawing guidelines, selection of the sustainable option, functional and stylistic interpretation of the elements according to the assumed aims, direct value-adding. This kind of new approach to the design process has to be seen as a contemporary need, a specific need in a globalized world, and aims at giving answers to specialists in search of value-adding elements of local tradition in their architectural and furniture projects.

The newly created instruments and methods for investigating, assessing and addressing critical vernacular architecture and furniture in national and European level contribute to a holistic vision of the vernacular phenomenon. A value-adding methodology of vernacular elements on European and national level was realized, comprising six stages grouped into two cycles, first with theoretical stages such as consultation of existing databases, decision making, drawing guidelines; the second cycle comprises the practical steps such as selecting the sustainable direction, functional and stylistic interpretation of the elements according to the aims assumed, direct value-adding through architectural and furniture projects.

The applicability of the proposed methodology in the field of architectural and furniture design in harmony with contemporary requirements was checked by a few projects: a sustainable housing unit and an innovative furniture ensemble designed according to the methodological steps that resulted from the research.

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BODY PRESSURE DISTRIBUTION ANALYSIS OF LAYERED FOAM SYSTEMS

Réka Mária ANTAL
University of Sopron, Simonyi Károly Faculty of Engineering, Wood Sciences and Applied Art
Bajcsy-Zs. street nr. 4, 9400 Sopron, Hungary
Tel: 0036 99 518 638, E-mail: antal.maria.reka@uni-sopron.hu

Péter György HORVÁTH
University of Sopron, Simonyi Károly Faculty of Engineering, Wood Sciences and Applied Art
Bajcsy-Zs. street nr. 4, 9400 Sopron, Hungary
Tel: 0036 99 518 638, E-mail: horvath.peter.gyorgy@uni-sopron.hu

Levente DÉNES
University of Sopron, Simonyi Károly Faculty of Engineering, Wood Sciences and Applied Art
Bajcsy-Zs. street nr. 4, 9400 Sopron, Hungary
Tel: 0036 99 518 231, E-mail: denes.levente@uni-sopron.hu

Abstract

This article presents the results of an experimental research based on the measurement of compression stresses of differently layered polyurethane foam structures with various foam sheet densities. For data acquisition the Tekscan's CONFORMAT body pressure distribution measuring system was used. The three-layered foam sheet system was loaded by a standard seat loading pad with four compression forces. Pressure distribution maps were recorded for all foam sheet variations and the contact surface and peak pressure values were determined. Based on the research results the comfort of the stratified cushion like structure was evaluated. According to the pressure distribution maps the homogenous foam layers with densities of 25kg/m^3 showed lower contact areas but a more uniform pressure distribution and lower peak pressure values. The heterogeneous stratification led to various contact surface areas and pressures, however, at 250N load force the 35-35-43kg/m^3 or 35-35-35kg/m^3 variations offer the highest contact surfaces and lowest peak pressures. At 500N load the 35-43-35kg/m^3 combination offer the best option, at 750N load the 35-25-25kg/m^3 and at 1000N the 43-25-25kg/m^3 variations have the optimal pressure distributions. Placing a transient foam layer between high and low density foam sheets did not indicate any significant attenuation.

Key words: *polyurethane foam; layered structures; comfort analysis; body pressure distribution; chair comfort.*

INTRODUCTION

Sedentary life-style of the human kind is getting more and more common leading to several musculoskeletal disorders. The increase of the seated occupations and sitting times raise the risk factors in the development of low back pain and other cardiovascular problems (Vink and Hallbeck, 2012). Consequently, seating devices, i.e. chairs must provide more comfort to diminish the negative consequences of prolonged sitting. From ergonomics point of view, the high comfort is related to the well-being, safety feeling and healthy sensation of the chair users. However, the enumerated subjective evaluation criteria can be fulfilled mainly by objective design specifications. Consequently, the sitting comfort of a seating furniture is the combination of the embedded materials, construction and other design factors like dimensions, tilt angles, etc., which may either, add to or detract from the comfort of the finished product. Construction of upholstery, shape and hardness of the sitting surface are included also into features, which determine the sitting comfort (Kapica and Grbac 1998). The characteristics of upholstery are important for comfort and proper distribution of pressure nevertheless, the basic factor of contemporary comfort is the specific pressure to the body, not the softness of the seat. This pressure is smaller when the contact surface of the human body is larger (Ergié 2002, Grbac and Ivelić 2005). Other scientific articles focused also on revealing the relationship between sitting comfort and design specifications with the aim of reducing the discomfort of chair users. For example, Manfield *at al*. (2015) analyzed the discomfort in vehicle seats and concluded that foam composition can have significant implications on people undertaking journeys of long duration (more than 40 min. in the conditions tested). Comparing different foam types, they determined the difference in overall seat discomfort. Small changes to foam composition were shown to affect the overall discomfort in the seat. According to Vlaović *at al.* (2010) there are major differences among the

materials of seat upholstery and their constructions. They conducted an experiment to determine the comfort index (support factor) of chairs obtained from elastic characteristics of materials in the seat of chair. In the result of examination of mechanical characteristic of chairs with polyurethane foams, a better comfort index has been noticed for chairs where in the subjective test evaluated as uncomfortable (Vlaović *at al.* 2010). In another study analyzing different types of seats, they concluded that the chair with molded PUR foam is significantly more comfortable than the chair with springs, but statistically it does not differ significantly from the chair with PUR foam cushion (Vlaović *at al.* 2016). According to Vink and Lips (2017) the form of the area contacting the body and the softness of this area influence the contact area between the body and the product. The pressure sensitivity of the skin and underlying tissue also plays an important role in the comfortability (Fig.1). Moreover, in seating design to create a comfortable seat it is important to define the foam characteristics of the seat pan or the flexibility of the material underlying the foam (Vink and Lips 2017).

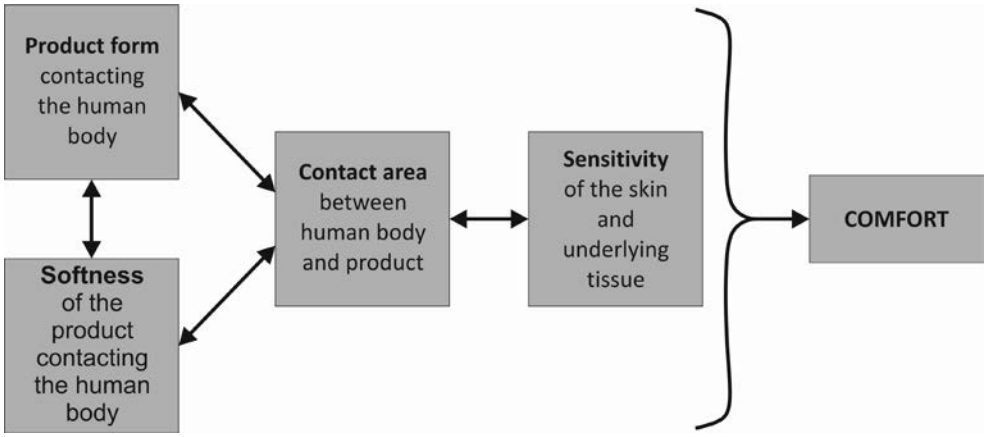


Fig.1.
Modelling the characteristics which influence comfortability (source: Vink and Lips 2017).

According to US Patents there are some inventions relates to the field of cushions. Rose *et al*. (1994) disclosed a type of cushioning device in 1994. Their seat cushion was made of layers of polyurethane foam, each layers having a different density. Discrete orthogonal support system was invented by John D. Clark (2007). The stratified cushion assembly can be used to support the human body under various conditions and includes alternating strata of supportive material. Each stratum have a different compression modulus and strata of visco-elastic memory foam and open cell polyurethane foam are attached together (Clark 2007).

Other studies indicate that pressure measure seem to be a highly associated and objective method for the quantification of subjective comfort or discomfort (De Looze *et al.* 2003, Mergl 2006, Verver 2004). According Brienza *et al*. (2001) interface pressure mapping is an accepted method used by researches to evaluate pressure redistribution in seating. According to Zemp *at al.* (2015) pressure distribution measurements of the seat pan and the backrest are one of the most common objective methods to analyze or compare different sitting positions. Hochmann *at al.* (2002) evaluated four different seat pressure-mapping systems (FSA, Xsensor, Tekscan ClinSeat, Novel Pliance) concerning their accuracy, linearity and hysteresis. Pressure mats are very sensitive to differences in the surface area properties of the analyzed product, therefore it is very important that sensor mats to be calibrated for the corresponding surface (Zemp *at al.* 2016). According to Zemp *at al.* 2016, the pressure mat should be calibrated for every chair, as far as the pressure distribution measurements are influenced by different material properties and geometry of the padding material. Jackson *at al.* (2009) have shown that the best performing cushion had a layered structure made of approximately 25 mm of Confor C47 foam with an overlay of approximately 13mm of Confor C45 (Confor is a medium density, open-celled polyurethane foam). They used a Tekscan type 5315 sensor mat for measuring the pressure over the seating and measured 5 different viscoelastic foam cushions that had not previously evaluated for gliders. The using method is suitable for comparing the comfort of different foams and combinations of foams. According to Horvath *at al*. (2016) the seat cushion can reduce up to 50% of the peak pressure of the sitting bones' (Ischial Tuberosity) zone. Not just the pressure mapping systems are used to define the comfort of seating furniture but the applied finite element model too, to simulate the contact interaction between human body and seat under different support conditions (Guo *at al.* 2016). Mohanty and Mahapatra (2014) analyzed the effect of cushion material properties and demonstrated that the use of right kind of foam for seat cushion and thickness

can substantially reduce the stress level at ischial tuberosity. According to design engineers from Advanced Design & Analysis Division, IDOM and Centro Tecnologico Grupo Copo developed a virtual environment for foam seat testing. The study focused on the assessment of numerical simulations, primarily related to comfort using Abaqus Finite Element Analysis (FEA). The experimental results demonstrated that comfort parameters can be successfully simulated numerically and FEA is definitely a valuable tool for assessing seat designs for comfort.

OBJECTIVE

Based on the reviewed professional literature we could conclude that few research results exist related to the influence of chair foam cushions and sitting comfort. Therefore, the main objective of the present research was to evaluate the pressure distribution of layered polyurethane foam systems with foam layers having different densities and elasticities, using a body pressure distribution measuring system and a standard loading pad. A second objective of this research was to determine the optimal arrangement of a 3-layered system assuring the best comfort of the chair users.

MATERIAL, METHOD, EQUIPMENT

Polyurethanes are generally considered to be the most versatile among plastics, because of the variety of properties they possess. They are foam materials that can be manufactured at varying degrees of density and softness. Depending on the initial raw material they can be hard or soft, elastic or rigid. These foams have a wide variety of applications, for example modern homes and offices would be far less comfortable without polyurethanes. Flexible polyurethane foams are soft, yet provide good support, durable, and maintain their shape therefore are preferred as filling materials for seating cushions and mattresses and can be produced to the density required by the manufacturer. Their versatility allows designers to use the full scope of their imagination when creating new products.

For our research purposes open cell polyurethane foams with different densities produced by Eurofoam Hungary Ltd were used. The selected foam types frequently used by Hungarian upholstery furniture producers belong to the Eurofoam Classic family, class N and R. The properties of the selected foam types are presented in Table 1:

Table 1

Characteristics of the PUR foams used in research

Type of PUR foam	Color	Density (kg/m^3)	Compression hardness, kPa (DIN 53577)	Tensile strength, kPa (DIN 53571)
Normal, N2538	Violet	25	3,8	110
Normal, N3530	Grey	35	3	90
Flexible, R4342	Green	43	4,2	100

From the selected foam types 600mmx600mm sheets with a thickness of 20mm were prepared and 3-layered structures arranged in every combination of the foam types, resulting in 27 experimental setups, totally. The layered structures were loaded with an anatomical seat loading pad according to standard EN 1728:2012.

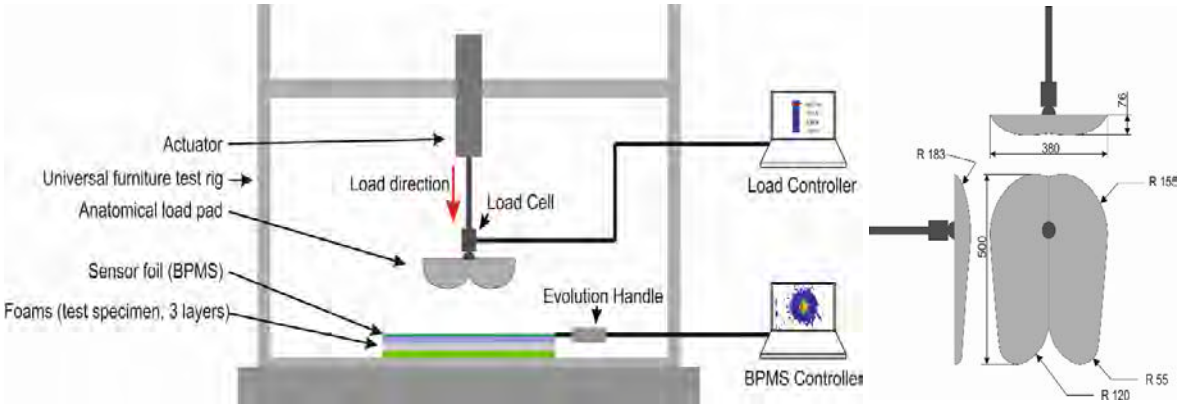


Fig. 1.
The schematic principle of measurements and the geometry of the loading pad.

The load intensity was set at four levels, 250N, 500N, 750N, and 1000N respectively, the maximum set load values were attained in 3 seconds. 108 measurements were made totally using the Tekscan's Body Pressure Measurement System (Conformat) with pressure sensitive foils size of 488×427mm containing 2016 pressure points with pressure range of 0-350mmHg, and accuracy of + / - 3.5mmHg. The computerized data recorder provides a real-time picture of the pressure distribution. The schematic principle of measurements is shown in Fig. 1. Before using the BPMS measuring system the pressure sensor foils were calibrated with the help of a vacuum pump. After calibration the pressure maps of layered foam structures loaded with four compression force values were collected and analyzed with the software delivered with the system (BPMS Research 7.20) in the form of image (.FSX or .jpg) or short (0-200 s long) video files. On the recorded pressure maps, the contact surface area and peak pressure values were determined.

Fig. 2. displays the experimental setup with pressing test rig, computer control and loading pad:

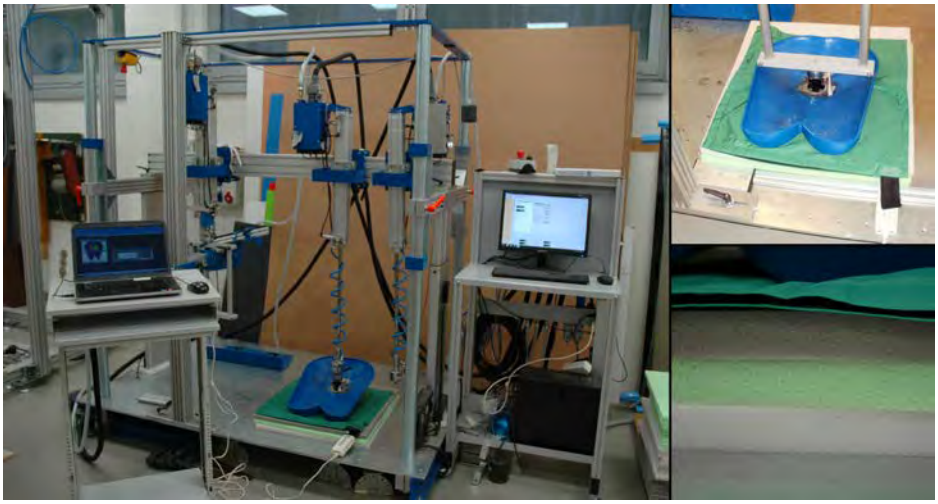


Fig. 2.
Laboratory test setup with the anatomical seat load pad and layered foam structure.

RESULTS AND DISCUSSION

The pressure distribution maps were recorded for all foam layers' variations and analyzed based on the contact area, pressure dispersion, pressure change intensity of different zones etc.

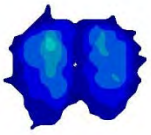
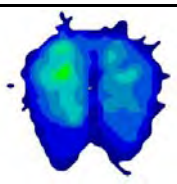
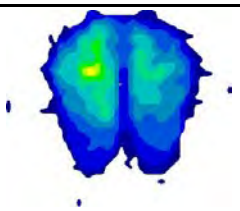
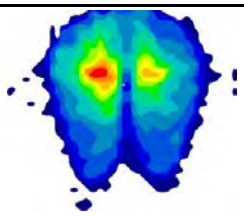
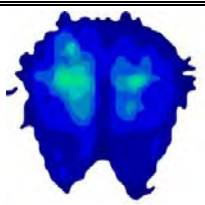
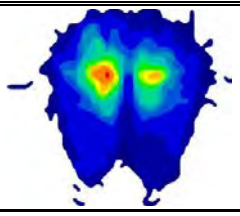
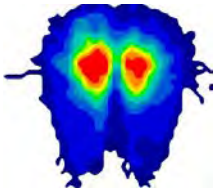
Pressing force, N	**250**	**500**	**750**	**1000N**
Foam density: **25 kg/m^3** (all 3 layers)				
Contact area, cm^2	399,48	669,93	946,58	1 095,22
Peak pressure, N/cm^2	1,12	1,50	1,98	2,59
Foam density: **43 kg/m^3** (all 3 layers)				
Contact area, cm^2	516,13	928,00	1 083,87	1 168,51
Peak pressure, N/cm^2	0,73	1,39	2,44	2,68

Fig. 3.
Pressure distribution maps of various pressure forces.

In Fig. 3. the effect of load intensity on homogenous layered foam structures is represented. The comfort normal foam type with lower density interact less with the load pad than the higher density comfort elastic foam however, the pressure distribution and pressure contours show more even dispersions mainly at higher loads. The lower density homogenous foam system induces lower shear stresses on the chair user assuring a higher comfort. The higher density foam compacts more therefore the deformation extends on a higher surface. A moderate asymmetry between right and left sides was observed.

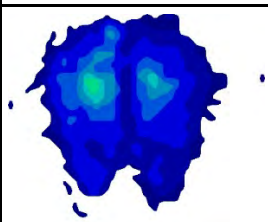
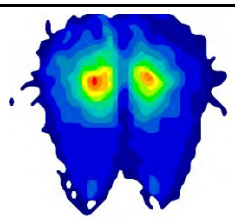
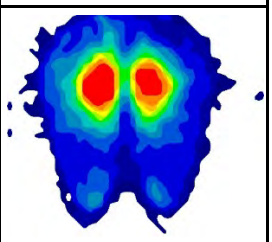
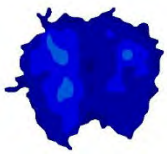
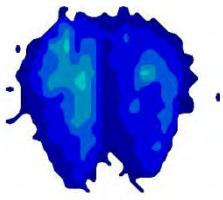
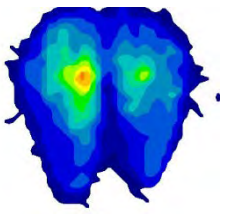
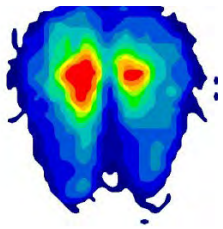
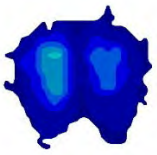
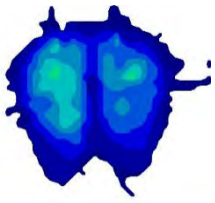
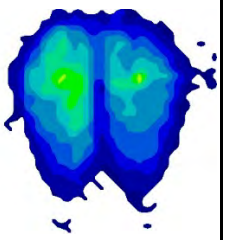
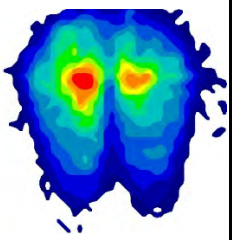
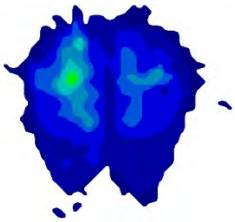
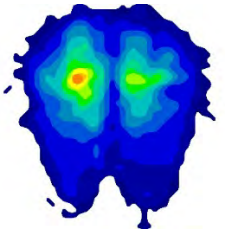
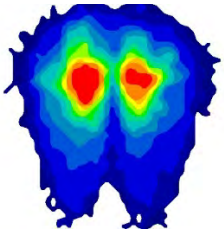
Pressing force, N	**250**	**500**	**750**	**1000N**
Layered structure: **25-43-43 kg/m^3**				
Contact area, cm^2	563,61	905,29	1 058,06	1 125,16
Peak pressure, N/cm^2	0,76	1,39	2,45	2,68
Layered structure: **25-25-43 kg/m^3**				
Contact area, cm^2	498,58	837,16	1 011,61	1 105,55
Peak pressure, N/cm^2	0,77	1,11	2,18	2,66
Layered structure: **43-25-25 kg/m^3**				
Contact area, cm^2	474,84	764,90	985,80	1 143,74
Peak pressure, N/cm^2	1,03	1,30	1,63	2,62
Layered structure: **43-43-25 kg/m^3**				
Contact area, cm^2	557,42	924,90	1 019,13	1 217,03
Peak pressure, N/cm^2	0,90	1,54	2,32	2,69

Fig. 4.
Pressure maps of different layered structures.

Fig. 5 presents the pressure distribution maps of heterogeneous structures when two layers of same low or high density foam sheets are combined with a third one of opposite density. In the case of high-high-low and low-low-high stratification the contact areas are very similar at any loading forces, but if the lower density foam layer is placed on the bottom the pressure is more uniformly distributed, the pressure gradient steepness is smaller. From seating comfort point of view, we found the best solution when a 43kg/m^3 density foam layer is placed on two 25kg/m^3 density layers.

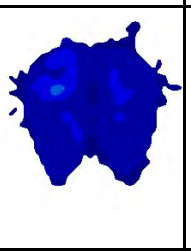
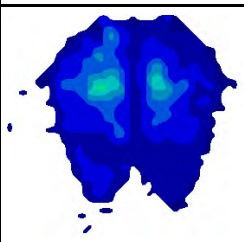
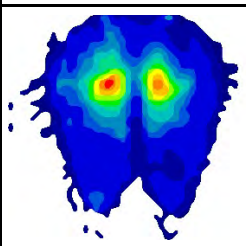
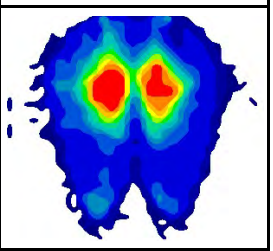
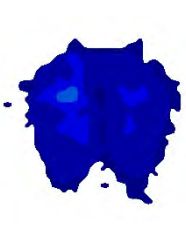
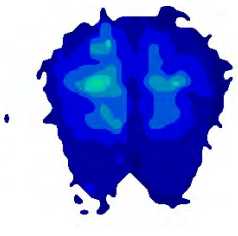
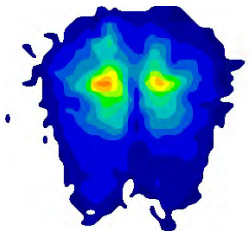
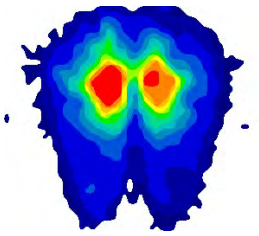
Pressing force, N	**250**	**500**	**750**	**1000N**
Layered structure: **25-35-43 kg/m^3**				
Contact area, cm^2	595,61	937,29	1 076,64	1 165,42
Peak pressure, N/cm^2	0,65	1,26	2,48	2,69
Layered structure: **43-35-25 kg/m^3**				
Contact area, cm^2	624,51	970,32	1 133,42	1 235,61
Peak pressure, N/cm^2	0,74	1,27	2,27	2,69

Fig. 5.
Pressure maps of layered structures with transient densities.

The influence of a transient layer with a density of 35kg/m^3 placed between the low and high density layers is shown in Fig. 5. The contact areas are maximum at all load values when the high density foam layer is placed on top, but the peak pressures are higher and pressure distribution is worse compared with the 43-25-25kg/m^3 structure. From pressure distribution perspective, the effect of transient layer is marginal. Loading the three layered foam structures with a load of 1000N lead to a peak pressure of 2,69N/cm^2 in almost all loading cases which demonstrates that the loading pad compressed the foam layers completely and was supported by the test rig's hard base. This means that a 60mm thick layered foam system using foams with 25, 35, 43kg/m^3 densities cannot attenuate completely the pressure exerted by a person of 100kg weight. The maximum peak pressure at 750N load was 2,61N/cm^2, and obtained by using a homogenous mid density foam structure. At 500N the highest value was 1,58N/cm^2 on a structure composed by foam layers having 35-35-25kg/m^3 density values. At the lowest load rate the peak pressure was 1,12N/cm^2 when a 25-25-25kg/m^3 configuration was used.

CONCLUSIONS

The comfort related pressure distribution on layered foam cushions was measured using a body pressure measuring system. A three-layered structure was prepared using upholstery foam types with three densities, and all possible variations. An anatomical loading pad imitating the human buttock loaded the layered structure using four pressure forces. Pressure distribution maps showing the compression load intensity, the pressure gradient, peak pressure values and contact areas were analyzed and evaluated from sitting comfort point of view. When homogenous foam structures were used the lowest density foams shown not just smaller contact areas indifferent of the selected loading forces, but a more even compression stress distribution. In the case of heterogeneous stratification, the lowest pressure values were determined for the high-low-low density configuration. Placing a transient foam layer between high and low density foam sheets did not indicate any significant attenuation. The lower peak pressure values do not always relate to a higher contact surface or even

pressure distributions. Even though a hard anatomical loading pad was used for measurements a more or less accentuated antisymmetric pressure distribution between left and right zones were observed. Based on the recorded surface contact areas and pressure distribution maps an optimal layered cushion can be developed. For an average user a layered system composed by a 35-25-25kg/m^3 density foam sheets or 43-2543kg/m^3 sheets assure a more uniform and wide pressure distribution leading to a higher comfort sensation.

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A VISION OF THE HUMAN BODY AS A SEATING CONCEPT

Ioan MUSCU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: ioan.muscu@unitbv.ro

Marina CIONCA
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: marinacionca@unitbv.ro

Biborka BARTHA
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: bartha.biborka@yahoo.com

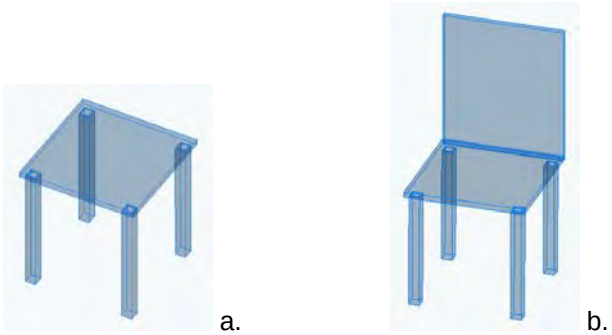
Abstract

The paper presents the results of studies and research aimed at identifying general and particular features of the chair design process. Three highlights of 20th century chair design were chosen as study cases, due not only to their innovative contribution in areas like construction, materials and technology, but also to their iconic role in setting new forms and new concepts that remain still up to date. The analyses and investigations reach beyond the obvious structural, construction and ergonomic characteristics and discover the subtle links that design creativity seems to develop with the arts, with the world of archetypes, signs and symbols, with the postures of the human body, that become roots of the impulse to design a Chair. Rarely, outside the world of design, specialists agree to confirm the strong contribution of sensorial perception of forms and meanings in the design process. The discovery of possible archetypes, allusions and/or metaphors regarding postures of the human body, form becoming a logo of sitting, are factors that allow the configuration of new investigation instruments that generate a fresh impulse to understand and value creative thinking.

Key words: *chair design; concept; symbol; sensorial perception.*

INTRODUCTION. CONCEPTUAL STRUCTURE OF A CHAIR

To define and understand a chair from a structural viewpoint requests the identification and individual naming of its elements and/or subsequent components. Hence the chair is supposed to have the following elements, grouped in three components: the supporting component (supporting legs); the seating component (the seat plane or frame); the backrest component (the chairback plane or frame).



a. b.

Fig. 1.
Standard morphology of a seating object. a - stool; b - chair.

These groups of elements are sufficient to answer the main functional requirements, namely to support, to prop up the human body in its sitting posture. They are able to define in a simple way a structural archetype, a so-called „icon" (Fig. 1). These icons are visually recognizable and we call them by a generic name – seats; their derivatives are: stool, chair (with a backrest), chair with a

backrest and armrests, and so on. The design process of a chair may ask for this analytical approach level, supported usually by dedicated creation methods, for example: morphological analysis, functional analysis, comfort analysis, aesthetical analysis. If we look at this product system we notice that a chair does not express only its related components in a formal whole, answering one function, favouring it in particular. The offer is complex and includes chair images and versions that are able to communicate also something else, they show a „personal" wish to manifest themselves and attempt to disclose their „productness" through another kind of language. The acceptance of this new reality leads to the assertion that the creation method of a chair is multilayered, reconsidering and restructuring itself permanently, according to the context and the spirit of time.

OBJECTIVES AND METHOD

The first investigations aimed at highlighting constant structural values when approaching the design of a chair. This favoured the understanding of the moment when a new structural approach of a chair emerged, and in this context, the possible causes and social-historic combinations of circumstances that favoured or strongly helped define the new structural concept that was approached - the cantilever concept and its consequences for chair design, were presented.

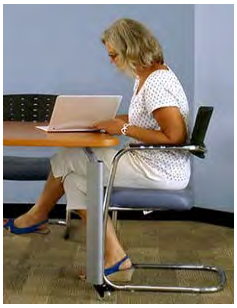
The theoretical research was developed along two main paths: 1. defining the elements and the objective characteristics that determined the conceptual diversification of a chair: a. analysis of the significant historic period and identification of the apropriate characteristics and features regarding chair design; b. the coming out of new materials to be experimented for furniture; c. new searches and solutions for technical and technological developments; d. evolution/transformation of the sitting positions; 2. defining elements and characteristics with a relevant inspirational potential, able to trigger ideas for a new structural concept. In this regard it became necessary to understand the chair as a bearer of senses and means of visual communication, to identify new visions and ideas beyond the strict manufacturing process, to discuss about the personality of the designer and the definition of the designed object as a „phenomenon on show".

The experimental analysis was carried on through direct collection of data, measurements and visual investigations regarding dimensions, angles, sitting positions, followed by sketching and drawing. Nevertheless the final goal to achieve regarded the aspects of sensorial perception as a vital contribution to design spirituality.

ANALYSIS OF CHAIR DESIGN

Sitting Attitude and Posture

A chair has to satisfy the main functional requirement – to assure a reasonable seated position. The aspect of sitting expresses a certain posture. A serious problem is to make sure that the chair harmonizes the sitting posture with need to sit, functionally and qualitatively. At an axiomatic level, a certain posture favours the optimum fulfilling of a need, or a certain need asks for and defines a favourable posture. The images in Fig. 2 are thus edifying.


a.

b.

c.

d.

Fig. 2.
Relevant sitting postures.

The constructive and ergonomic characteristics, the quality of the materials and many others may become constant values, influencing one result or another. If we focus on the main segments of the human body in every sitting posture shown in Fig. 2 it is obvious that the the general aspect can be stylized and interpreted as an iconic sign that becomes a synthesis of the relationship between the position of the seated body and the form of the chair. The four situations from Fig. 2 are illustrated by the iconic signs shown in Fig. 3 and described in Table 1.

a. b. c. d.

Fig. 3
Structural icons of a chair.
a. icon of the classic chair concept; b. icon of a cantilever chair; c. icon of the Zig- Zag chair; d. icon for a chaise-longue.

The four icon models are to be interpreted as essentialized images of constructive-functional visions that define a chair typology. Based on these icon models we are able to identify a relationship of biunivocal determination between the constructive concept of a chair structure and the using of new materials and technologies in the manufacturing process. In some of the icon models we recognize certain influences or sources of inspiration from the area of the arts and/or crafts that are going to be also explored.

Table 1

Interdependence between necessity and definition of the seating furniture concept

Necessi-ty	Posture	Ergonomics	Construction	Materials	Intensity of manifestation	Duration of manifesta-tion
Work and activities that require movement	a-b-c	- sitting height correctly defined; - backrest; - freedom of moving arms and legs.	- high level of adjustment, adapting and transforming constructive elements to parts of the human body	- optimal qualities for the sitting comfort and for elements that are in contact with the human body	- high and medium intensity with a major influence upon the whole concept	- medium influence upon the concept
Rest and repose moments	b-c-d	- back-and head rest; - leg and arm rests	- does not require special adjustment of the constructive parts	- optimal qualities for the sitting comfort and for the elements that are in contact with the human body	- moderate intensity with medium influence upon the concept	- medium influence upon the concept
Rest and relaxa-tion	c-d	- the interior angle between the seat and the back over 100^0; - the entire chair inclined towards the back; - support for all parts of the human body	- high level of adaptation of constructive parts to the human body; - the chair typology migrates towards the easy chair	- high qualities for the sitting comfort and for the parts that are in contact with the human body	- high intensity with major influence upon the entire concept. Correlated with ergonomics and construction of product	- major influence upon the concept

Structural Deconstructions of a Chair

A function of an object, be it primary or secondary, may take various appearances or expressions. This is likely to happen because the design of an object, the idea, the image or its formal apparition may have different triggering causes and various sources of inspiration. How can we explain the two icon examples presented in Fig. 4?

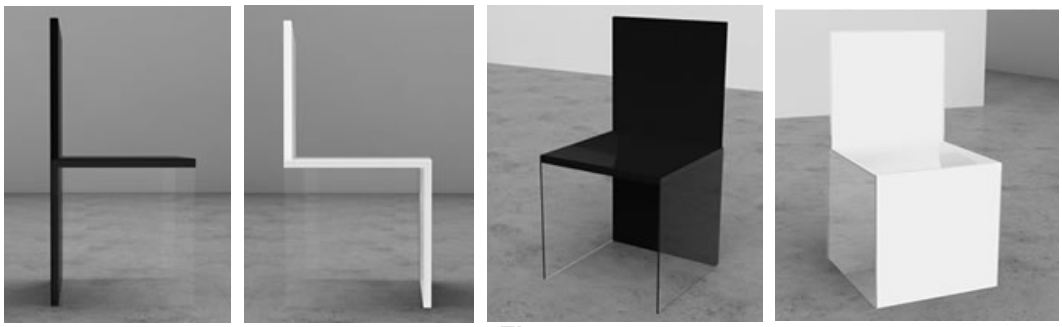


Fig. 4.
Structural iconography through morphologic stylization.

Certainly they cannot be the result of a functional and morphological analysis. The creative-experimental approach of these representations starts from the area of a sensorial perception of stimuli and presents a „possible" seating object that appeals to a plastic-expressive language and proposes a simple compositional play, a structural-formal composition expressed by opacity and transparence (material-immaterial), fluency or spatial suspension, sign/symbol or even logo.

Cui prodest - who benefits? The piece of furniture cannot be separated from an existing spatial context and in this case the object is composed and imposes itself in the same sense or in contrast with the expressed architectural environment. The morphologic stylization of a chair answers a need of clarification and ambiental synthetization through an expression of formal and structural purity. Aren't all of these characteristics and values of a contemporary environment?

CANTILEVER DESIGN

Forms of a Cantilever Concept

A cantilever chair is a chair whose seat and/or back are not supported by the classical arrangement of four legs, but instead is supported by a single leg or legs that are attached to one end of a chair's seat and usually bent in an L shape, thus also serving as the chair's supporting base. „Originally developed in tubular steel by the Dutch architect and furniture designer Mart Stam in the 1920s, soon after its invention the cantilever chair has found itself the centre of attention, inspiration and reinterpretation among some of the most prominent Bauhaus figures such as Marcel Breuer and Ludwig Mies van der Rohe. Later, in 1960s, Verner Panton has popularised the form by creating the now-iconic, curvilinear Panton chair which, at the time, was the first cantilevered chair made from a single piece of plastic. Since then, the 'chair without legs' has been revisited and refashioned innumerable amount of times by designers from across the globe", according to www.dailytonic.com.

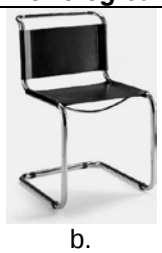
Chronological concepts - historical milestones				
				
a.	b.	*c.*	*d.*	*e.*
Mart Stam, Gas Pipe Chair, 1926,	Mart Stam, S 33, 1926	L. Mies van der Rohe, MR 10, 1926/1927	Marcel Breuer, Thonet B33 , 1927-1928	Alvar Aalto, Chair No31, 1930

Fig. 5.
Early chronology of cantilever chair design.

While interpreting the general form of the cantilever concept, a simplification of the ensemble of defining elements, a reduction of their number are noticed, in order to achieve a structure preponderantly rectangular, just like a continuous plane that is folding in space and describing a cursive, stable and elastic iconic direction (Fig. 5). The main defining features of a formal cantilever concept are: a. the general shape of the chair suggests a spatially folded plane; b. the general form expresses fluency, resistence and elasticity; c. utilisation of a single material for the entire structure; d. reduction of the number of elements; e. reduction of the object's weight; f. simple, undismountable construction; g. complete lack of decorative elements.

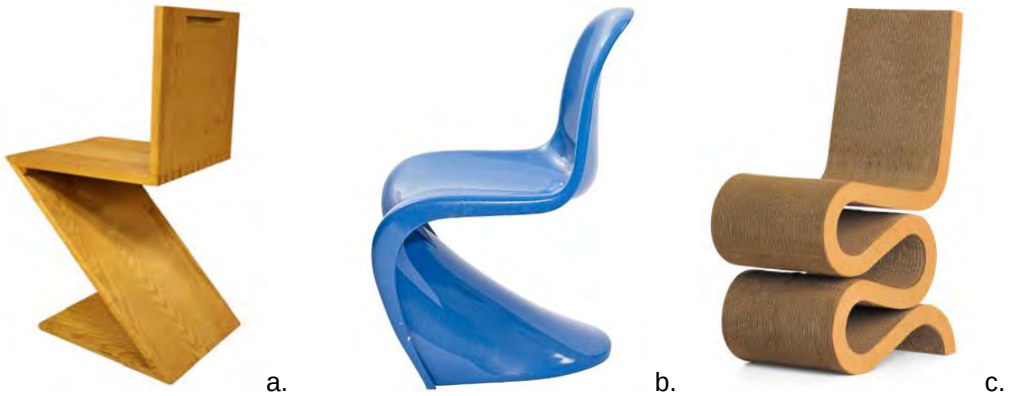
The dispute upon the tubular metal chair paternity includes the three great architects Mart Stam (1889-1986) Marcel Breuer (1902-1981) and Ludwig Mies van der Rohe (1886-1969). The orientation of a cantilever chair towards a wood structure happens with Chair No. 31 of the Finnish architect Alvar Aalto (1898-1976), in 1930.

Our analysis goes on with the cantilever concept that is tied up, in a ramified manner, to the Neoplastic inspiration.

Folding Plane – *Zig Zag* icon

The chairs that were chosen for the analysis are shown in Fig. 6 and develop chronologically in a period when technological innovation and conceptual thinking bring characteristics and values defined by: elementarism/ neoplasticism; rationalism; organic expression; technological performance; economic efficiency, specific requirements of modernity.

At least the first two chairs are conceptually and expressively tied up to the De Stijl movement, even if they are not made of the same material and do not belong to Neoplasticism, but to later style developments.



a. b. c.

Fig. 6.
The analysed chairs
a. Zig-Zag chair, 1932-1933; b. Panton chair, 1960; c. Wiggle chair, 1972
(after Cionca, 2014).

"De Stijl [1917-1931] encompasses an idea, a movement, a periodical and, ultimately, a visual language". (Dettingmeijer et al. 2010). Those were times of postwar austerity and later on finacial crisis. „Cheap" experiments with „lost and found" materials were meant to satisfy unassuming social needs, not pretentious aesthetic fantasies. According to Ida van Zijl "The Zig-Zag chair …is more intriguing than that other favourite of the Modernist avant-garde, the Freischwinger [cantilever by Stam, Breuer etc]. The cantilever chair, which was first investigated in the mid-1920s by designers including Mart Stam and subsequently elaborated by many others, is based on a cube with one of its sides missing. The Zig-Zag chair, by contrast, is a sigle line in space and therefore the ultimate symbol of Rietveld's work for many people. Rietveld characterized the form 'a little partition in space…It is not a chair but a structural joke'…Rietveld was…forced to to restrict himself to timber, but from various sketches and prototypes it seems he was searching for a form that could be punched from the material in single mechanical process and then folded into shape. He sketched a chair of sheet iron and produced prototypes using fibreboard, plywood with strip iron, and tubular steel. [But] the execution in four planks reinforced by slats and bolts ultimately proved to be the most stable and the simplest to produce" (van Zijl 2016).

The image transfer from a classic cantilever chair to the Zig-Zag icon is based on the following arguments: a. An idea of structural-formal restylization coming near the De Stijl concept; b. the „mimetism" as a creation instrument applied to usual sitting postures; c. expressions of new technical

and technological performances regarding joints and spatial bonds between the constructive elements; d. reduced transportation and storage room through stacking. In its general form and according to its surface development in space, the Zig-Zag chair, designed by the Dutch cabinetmaker and architect Gerrit Rietveld (1888-1965), looks closer to van Doesburg's vision of Neoplasticism than to Mondrian's.

The organic vision of the Panton chair

According to Hanne Horsfeld in „Innovation-Integration-Provocation. Seating by Verner Panton" (Von Vegesack et al. 2000) „In the 1960s, the impact of young protests and pop culture was felt in the field of design…The young generation wanted to liberate themselves from the convention and the "good taste" of their parents. Pop became the key word in fashion and music, characterizing a new, young and informal life style. …In design, the Sixties became a decade of plastic, foam and modular furniture systems. Important impulses came from the science fiction craze, which had taken hold in all areas of culture in the age of space travel….Panton's organic world of forms is inhabited by shapes reminiscent of plants, the human body or internal organs. Associations with the profile of the human body are also triggered by the cantilever principle, which Panton returned to again and again...The Panton chair…embodied the integration of a chair's individual functional elements – back, seat, base – in a coherent form…Panton expresses doubt about the constant striving for comfort and relaxation, being of the opinion that the most important thing must be to 'seek harmony between people and their surroundings'…Panton's seats are addressed to all the body's senses, feelings and fantasy… Located somewhere between Organic modernism and Pop, Panton can be described as an inventor of "Integral Modernism". Verner Panton (1926-1998), a Danish architect and designer, sets an example with his Panton chair for the dialogue between a new material with its particular technology. Also he expresses the organic character of form, triggered already in the interwar period. Its concept comprises a shell type ensemble made of fiber-reinforced resin, joyfully coloured, that can be described as having: a structural-constructive simplicity, organic expressivity, ergonomic form, industrial potential, reduced weight.

Waves and vibrations: the Wiggle chair

The Canadian-American architect Frank O. Gehry (1929 -) is known for his use of futuristic shapes and unusual materials for both his architecture and furniture. With his series "Easy Edges" from 1972, he succeeded in lending such everyday materials as cardboard a new aesthetic dimension. Although the Wiggle Chair appears unbelievably simple, it is incredibly robust and stable while feeling at the same time surprisingly soft to the touch. Sixty layers of cardboard are held together by hidden sticks with a fiberboard edging. Gehry named this material Edge Board: it consisted of glued layers of corrugated cardboard running in alternating directions, and in 1972 he introduced a series of cardboard furniture under the name "Easy Edges." The ‚Easy Edges' were extraordinarily sturdy, and due to their surface quality, had a noise-reducing effect in a room", as seen on http://www.design-museum.de.

Experiments that deal with new materials and new technologies find their best example in the Wiggle chair. It seems that the Wiggle has the desire to express images of physical phenomena concerning the propagation of waves or of sinusoidal oscillations, and least of all the icon of a classic chair. The concept shows the following attributes: structural-formal simplicity; the use of easily recyclable material-corrugated cardboard; innovative construction and technology; industrial capability; reduced weight; expressivity of movement and oscillation on the vertical direction.

Visual references as feasible conceptual ideas

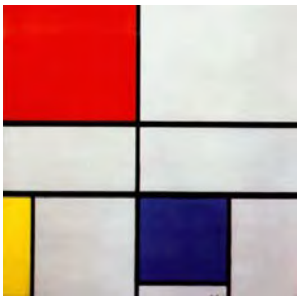
A creative leap happens when it is understood that everything that exists at a certain moment around us can start triggering stimuli or grow seeds of inspiration and awareness of various objects that await to express themselves or to be investigated. This becomes a viable observation when we agree with a visible conceptual similitude between situations, elements, examples that usually belong to different domains, directions and/or specialties. There is nothing mystical, codified or suspiciously persuasive in this, there is only an individual disponibility to recognize the existence of these impressions and the willingness to consider that sensorial perception has to be used as an instrument for knowledge enhancing, as efficient and valuable as reason itself.

An example: the structure and the overall form of the Zig-Zag chair can be easily identified as being supported by the philosophy and character of Dutch Neoplasticism. The transfer of these contents from the sphere of the art to that of furniture production is easily recognizable as a language of conceptual communication defined by formal purity, stylization and essentialization of the object's

structure until it reaches the symbolic level of understanding. Thus the similitude between art and product design defined as visual influence is continued (Fig. 9, Fig. 10).

In the case of these two significant images a „random resemblance" of the formal and compositional language can be noticed. This time the artwork is not the reference that has defined the chair concept – but the fact that both examples, even if they use different tools, talk about the same thing: the dynamics of movement. We can't ignore either of this image associations, with which we maintain the possibility to consider that the „illumination" in the simple creation process may appear through the mimetism pointed out by a random situation that is real. Can there be an affinity between these sitting postures and the Zig-Zag chair? It was asserted previously that possibly there are concepts and ideas triggering certain elements, but without insisting that it really happened.

a.



b

Fig. 7.
Geometric abstract art of the twenties: De Stijl
a. Theo Van Doesburg; b. Piet Mondrian.

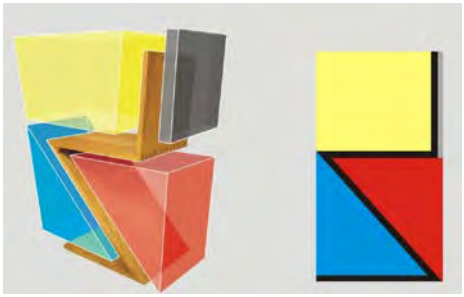


Fig. 8.
Structural-volumetrical composition of the Zig-Zag chair.

Fig. 9.
Nude Descending A Staircase by Marcel Duchamp.

Fig. 10.
Zig-Zag, remixed by Katerina Sokolova.

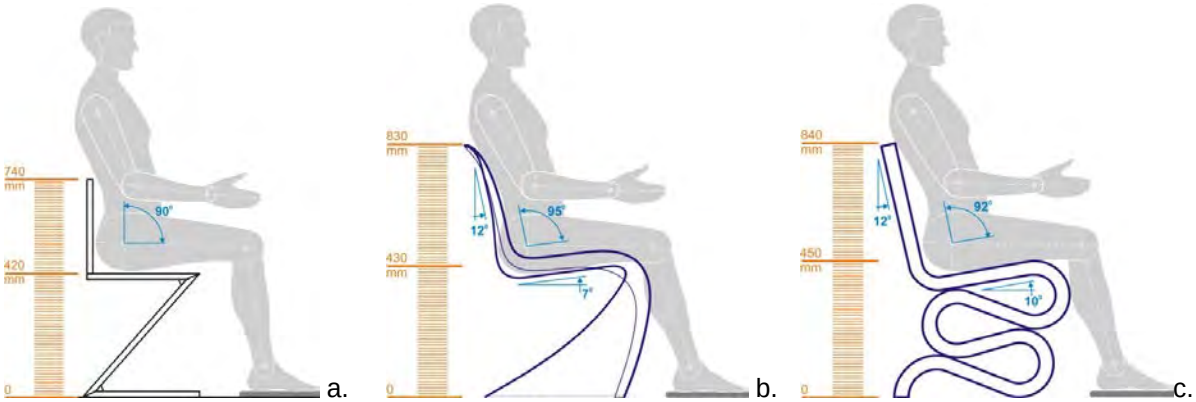
a.

b.

Fig. 11.
Visual references for a chair concept
a. Sitting posture – informal socializing position; b. Tubular crosslegged chair by Gerrit Rietveld, 1932-1933.

Sitting positions, dimensions and angles

The study of the sitting positions started from the necessity of comparing the dimensional and angular characteristics of the three chairs with those recommended by various specialists, preceded by the evaluation of the degree of comfort offered by each of them.



a. b. c.

Fig. 12.
Sitting positions. a. Zig-Zag; b. Panton; c. Wiggle.

The analysis of the sitting positions is shown in Fig. 12, where the position of the average (50% of percentile) human being is represented as sitting on the respectively scaled side view of each chair.

Table 2

Parameters of sitting positions on the three chairs, as compared with ergonomics data (Neufert 2004, Grandjean and Kroemer 1997, EN 1335-1:2000)

	Zig-Zag		***Panton***		***Wiggle***		***Recommended for side chairs***
Seat height in front (mm)	420	★	430	★	450	★	400-450
Seat depth (mm)		★		★		★	380-450
Total height of the chair (mm)	740		830		840		-
Seat angle	0^0		7^0	★	10^0	★	5-8˚
Seat-backrest angle	90^0		107^0	★	104^0	★	105-115˚
Backrest angles	0^0		12^0	★	12^0	★	13-15˚
Matches with the recommended parameters	**2**	★	**5**	★	**5**	★	

The results of this analysis are sinoptically presented in Table 2, together with a general evaluation of the chairs' ergonomics: the stars show the dimensional and angle parameters that coincide with those recommended by various authorities.

The concept of the Zig-Zag chair is not formulated to answer comfort requirements. It is a chair meant for short duration sitting. It respects the overall dimensions for this kind of product. The Panton and Wiggle chairs are examples that integrate comfort requirements. The Panton concept highlights the human body in its sitting position. The Wiggle concept shows its elastic attraction in order to enhance the comfort aura of the seated position. This attractivity results from the types of materials used and from the chair's general form.

Construction, Material, Technology

Figures 13 – 15 present details of construction and bits of the technological approach for the three chairs.

The Zig-Zag is a chair made of solid wood planks that are joined with classical dovetail and finger joints, enhanced by dowels. The flat components become a formal ensemble that looks like a folded plane. Although classical, the joints that allow glueing angles (40^0 - 45^0) are remarkably well chosen. Discrete triangular prismatic elements are inserted to increase the strength of the joints.

Regarding form development and communication through formal elements, the Zig-Zag is strongly pleading in favour of De Stijl – it is its conceptual determination. Mart Stam declared in 1934: "He [Rietveld] knows that what he needs to do is to find new materials with another and more simple form of assembly." And Mareike Küper said that „He tried a curved plywood version in 1938, for a living/dining room at Metz & Co showroom in Amsterdam, but this was a prototype" (Küper 1992). But it was too early a step in the molded plywood technology, that chair had to be reinforced with steel.

The Panton chair, Alexander Von Vegesack wrote, „represents a true synthesis of form, material and manufacturing technique" (Von Vegesack et al. 2000). The same author tells the story of its manufacturing: "Vitra decided to make it [the Panton chair] first of hand-laminated, fiberglass-reinforced polyester (1965)...Towards the end of the 1990s technical advances in processing plastics – in particular the refinement of injection moulding with the ability to achieve varying thicknesses in the shell wall - gave impetus to further development of the Panton chair…A model made of injection moulded polypropylene launched in 1999, finally achieved that the chair became, as Panton's wish was, "an inexpensive industrial product" (Von Vegesack et al. 2000). Monique Bucquoye fills in: "Panton had a stroke of genius in 1958 when he separated the concept of sitting from the stereotype concept: 'that thing on four legs'. In contrast with the Zig-Zag chair ... the Panton chair is not a manifesto but a functional object…In the wake of Pop-art, which was a frontal attack on institutionalized abstract art, pop design emerged with trendsetters" (Bucquoye 2002).

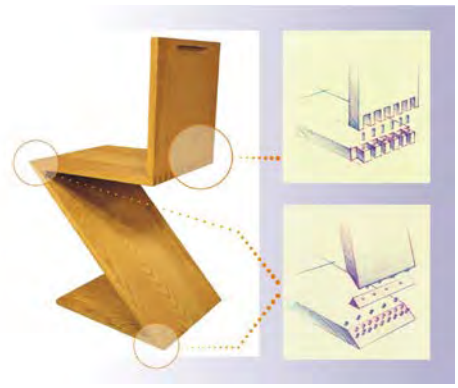


Fig. 13.
Zig-Zag chair. Original joints between the plane elements.

Fig.14.
Panton chair (polypropylene injection).

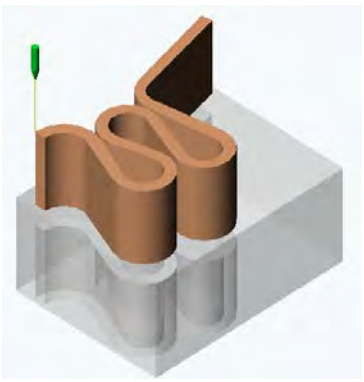


Fig. 15.
Wiggle chair (lasercut cardboard layers).

The formal elegance of the Panton chair is due to its similitude with the human body, as it was previously described.

The Wiggle chair is to be seen as the result of the most famous experiment in the field of furniture of the last 50 years. It succeeded due to its author's designer skills to elevate the „rank" of cardboard as a respectable material, from the level of a mildly appreciated packaging resource to the level of a beautiful, valuable, recyclable hence ecologic material. It requested an innovative technology, the lateral „ribbon" contour being laser cut up by a CNC in a volume of five dozen corrugated cardboard sheets. During the second part of the process, the „ribbons" are glued and transversally strengthened with 11 wooden sticks.

CONCLUSIONS AND DISCUSSIONS

The main structural elements of a chair are able to fulfill a double function, to support the human body in a sitting posture and sometimes to express a structural archetype which we called „icon". The latter eventually becomes a synthesis of the relation between the body position and the shape of the chair.

A chair may propose besides the image of a sign/symbol/logo also a simple compositional play, a structural-formal deconstruction expressed by its material/imaterial logic, its fluency and/or its spatial suspension.

The design process presupposes a high degree of availability from the creator to admit and consider the sensorial perception, which is a strong instrument of knowledge and action, as efficient and precious as reason itself. We should accept a visible conceptual similitude between certain situations, elements, example models that usually belong to different areas, directions and specialties.

The mission of the Zig-Zag chair is to be an experiment and an icon for a vision that is able to materialize itself simultaneously in the arts and in the industry. The Panton and Wiggle chairs are exceptional examples for illustrating the response to comfort needs, but they are much more: the former evokes a seated human silhouette, the latter describes, by its form and structure the physical phenomenon of elastic movement.

The analysis of the three chairs aimed to define them as highlights of chair design, asserting new visions through their form and functional-expressive structure. The historic reverberation of their concepts continues to bring new forms, new solutions and new technologies in the contemporary world of industrial design.

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EFFECT OF HEAT TREATMENT ON COMPRESIVE AND TENSILE STRENGTH OF END TO EDGE BUTT JOINT

Sergiu GEORGESCU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: sergiu.georgescu@unitbv.ro

Bogdan BEDELEAN
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: bedelean@unitbv.ro

Abstract

The effect of heat treatment on the compressive and tensile strength of end to edge butt joint is analysed in this work. The joints were prepared from both untreated and heat-treated ash (Fraxinus excelsior) wood. The end to edge butt joint made of heat-treated wood has a lower resistance both for compression and tensile strength, compared with joints made of untreated wood. The length of dowel has a bigger influence on compressive and tensile strength of joints than the distance between dowels and the ratio of dowel penetration in the main part of joint. An optimal solution to place the dowels is suggested both for the joints made of heat-treated wood and joints made of untreated wood.

Key words: *end to edge butt joint; heat-treated wood; ash; tensile and compressive strength; optimisation.*

INTRODUCTION

The joints used in the manufacture of wooden products must be able to take over, to transmit and to support the load required by their use. One of the most used joints in the construction of furniture is end to edge butt joint (Fig. 1). This type of joint is preferred for the easy of their processing (Negreanu 2003).

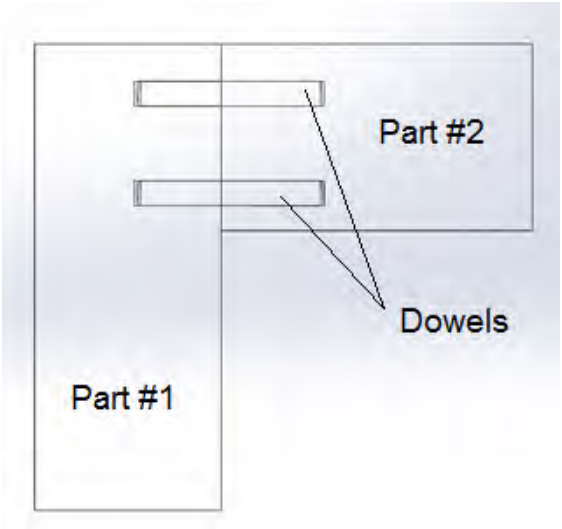


Fig. 1.
End to edge butt joint.

End to edge butt joint sizing is based on the existing recommendations in the literature (Curtu 1988, Cismaru 2009). These recommendations are based on studies that have been developed for solid wood joints, where wood has superior mechanical properties compared to heat-treated wood, whose main disadvantage is the reduced mechanical strength. This disadvantage can be reduced by appropriate sizing of joints used for manufacturing the products (Kuzman et al. 2015).

In order to obtain a proper design of a product made of heat-treated wood, it is necessary to know how much the strength of end to edge butt joint made of heat-treated wood is reduced compared

to untreated wood joint. To the best to our knowledge, there is lack of studies that deal with this topic. Also, there is a lack of studies regarding the sizing of end to edge butt joint made of heat-treated wood. This kind of studies could be useful to designers in order to establish an oversizing coefficient and to figure out the optimal dowel length, the distance between dowels and the ratio of dowel penetration in the main part of joint (see part #2 in Fig.1).

OBJECTIVE

The main objective of the present research is to figure out the behaviour of end to edge butt joint made of both heat-treated and untreated wood under the compression and tensile strength tests. Also, the objective is to figure out the optimal dowel length, the distance between dowels and the ratio of dowel penetration in the main part of joint.

MATERIAL, METHOD, EQUIPMENT

The material used in this research was untreated and heat-treated ash (*Fraxinus Excelsior*) boards. Some technological steps were followed in order to obtain the end to edge butt joint, as follows: drilling the wooden elements, gluing and jointing the parts and joints conditioning. Before gluing, the parts were selected and arranged according to the experimental plan (Table 1).

Table 1

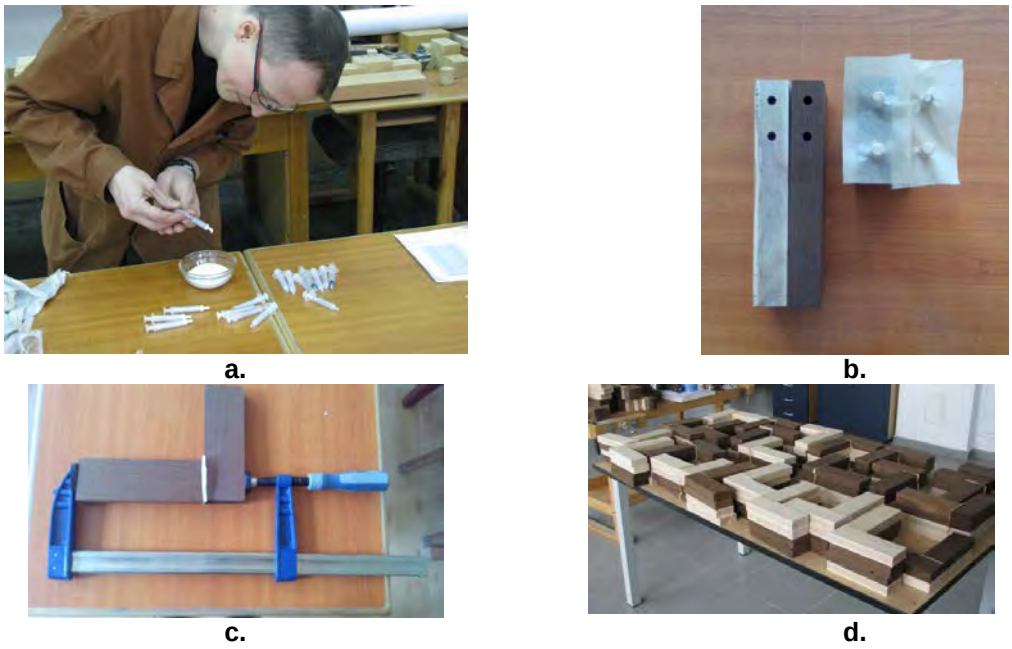
The experimental plan used in the present research

Configuration	Independent variables					Dependent variables	
	Distance between dowels (X_1), mm	Hole depth in the parts of the joints (X_2), mm			Dowel length (X_3), mm	Breaking compression force (Y_1), N	Breaking tensile force(Y_2), N
		part #1 of joint	part #2 of joint	Ratio of dowels penetration in part #2			
1	16	15	15	0.5	30	788	2330
2	32	15	15	0.5	30	1110	3740
3	16	21	9	0.7	30	1010	2540
4	32	21	9	0.7	30	1040	2180
5	16	30	30	0.5	60	2250	4990
6	32	30	30	0.5	60	2060	4460
7	16	42	18	0.7	60	2610	4730
8	32	42	18	0.7	60	1970	4200
9	24	27	18	0.6	45	2620	5420
10	24	27	18	0.6	45	2490	4510
11	24	27	18	0.6	45	2780	6120
12	24	27	18	0.6	45	2460	4610
13	24	27	18	0.6	45	1990	4460
14	16	27	18	0.6	45	3130	5350
15	32	27	18	0.6	45	3460	4970
16	24	23	22	0.5	45	2960	7460
17	24	32	13	0.7	45	2120	3580
18	24	18	12	0.6	30	1180	3190
19	24	36	24	0.6	60	3030	6310
20	24	27	18	0.6	45	2990	5930
21	24	27	18	0.6	45	2690	5880
22	24	27	18	0.6	45	2420	4450
23	24	27	18	0.6	45	2340	5700
24	24	27	18	0.6	45	2300	4620

Adhesive consumption rate was 350g/m^2, according to Negreanu (2003). In order to find the area of each hole, the SolidWorks software was used to 3D modelling of various holes depth, according to experimental plan. The quantity of adhesives needed to be applied in each hole was calculated by multiplying the adhesive consumption rate by area of each hole. The adhesive was applied by means of a 2ml syringe (Fig.2).

In order to obtain a good adhesion, after applying the adhesive, the parts waited a period of 10 minutes long before jointed. The possible influence of adhesive excess on the strength of joint was

limited by separating the parts of joint by applying wax paper (Fig 2b). The parts of joint were pressed after jointing in a screw clamping device (Fig. 2c) and were conditioned for two weeks (Fig. 2d).



a. **b.**

c. **d.**

Fig. 2.
Dispensing the adhesive (a), applying the wax paper (b), pressing the parts of joint (c) and conditioning the joints (d).

The mechanical testing of joints was performed on the universal testing machine Zwick Roell Z10. The load was applied at a constant speed of 3mm per minute until a significant separation between the two parts occurred (Kuzman et al. 2015). The value of the maximum breaking force was recorded for each tested specimen. The joints were tested both for compression and tensile load, as it is recommended in the literature (Fig.3). The devices were especially designed for this kind of test.

RESULTS AND DISCUSSION

The results show that most of the heat-treated joints have a lower compressive strength than untreated joints (Fig. 4). However, there are some exceptions for the configurations # 1, 4, 6, 12, 13, 18 and 24 (Table 1). Heat – treated wood joints has generally lower tensile strengths than untreated wood joints (Fig.5). The exceptions are configurations # 5, 6 and 15 that have higher tensile strengths than untreated wood joints (Table 1).

The heat-treated wood joints have a lower resistance than untreated wood joints values both at compression and tensile strength, respectively 13% and 21%. The percentage of reduction in the strength of the end to edge butt joint was calculated based on the central configuration (X_1=24 mm, X_2=0.6 and X_3=45 mm) that was imposed by the experimental plan (Table 1). The central configuration was repeated ten times.

a. ***b.***

Fig. 3.
Testing of end to edge butt joint at compression (a) and tensile (b).

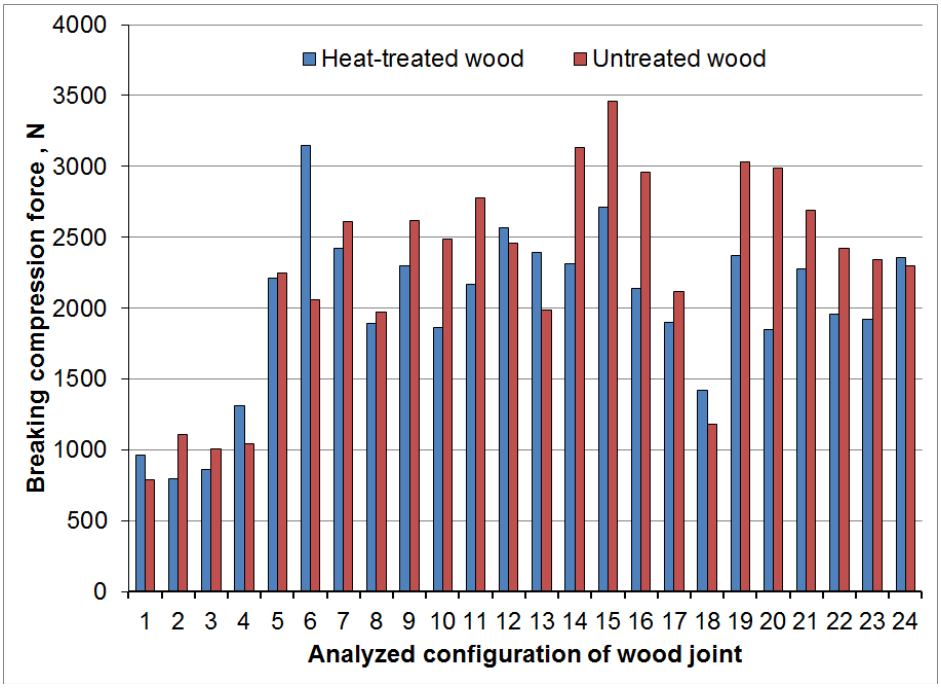


Fig. 4.
Breaking compressive force for end to edge butt joint made of heat treated and untreated wood.

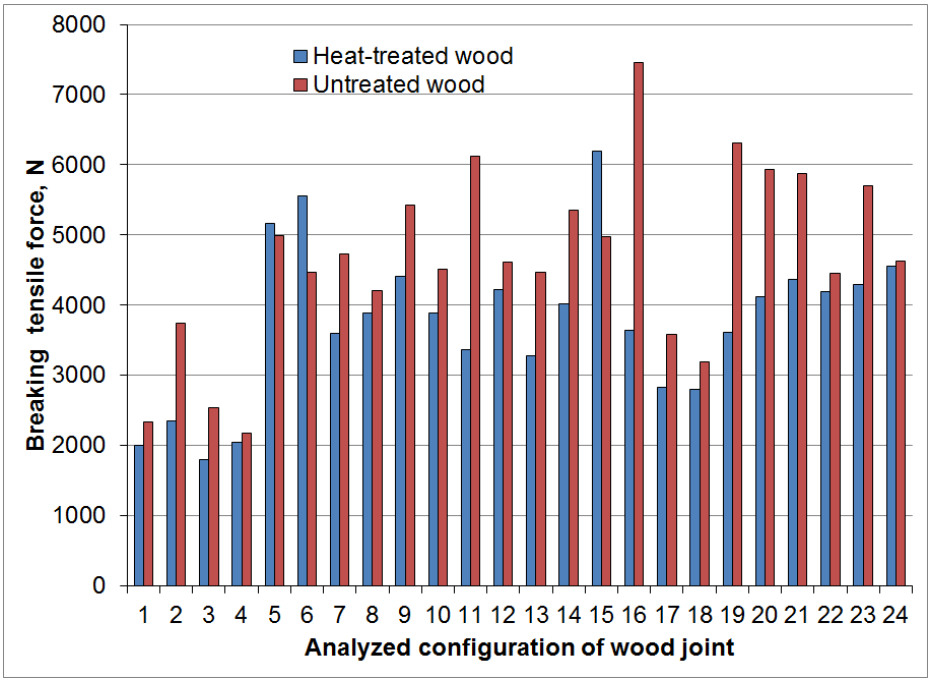


Fig. 5.
Breaking tensile force for end to edge butt joint made of heat treated and untreated wood.

Mathematical models (multiple regression equations) were established based on the experimental results by using the Design-Expert Version 9 – Stat-Ease. The models describe the relationship between independent variables (the distance between dowels, ratio of dowel penetration in the main part of joint and the length of the dowel) and the dependent variables (breaking compression force and breaking tensile force). The models were developed both for heat treated and untreated wood joints (Tables 2 and 3).

Table 2

Mathematical models that describes the relationship between independent variables and breaking compression force

Type of joints	Form of presenting the equation	Obtained Equation	Coefficient of determination (R^2)
Heat-treated	Coded	$Y=2208.01+108.90X_1-87.90X_2+669.30X_3-105.87X_1X_2+16.38X_1X_3-182.62X_2X_3+204.96X_1^2-285.04X_2^2-410.04X_3^2$	0.82
	Real	$Y=-16751.86–66.84X_1+41980.55X_2+278.41X_3-132.34X_1X_2+0.13X_1X_3-121.75X_2X_3+3.20X_1^2-28503.79X_2^2-1.82X_3^2$	
Untreated	Coded	$Y= 2571.96 – 14.80X_1-41.80X_2+679.20X_3+395.23X_1^2-359.77X_2^2-794.77X_3^2$	0.80
	Real	$Y= -15718.15 - 298.27X_1+42754.68X_2+363.18X_3+6.17X_1^2-35977.24X_2^2 – 3.53X_3^2$	

Table 3

Mathematical models that describes the relationship between independent variables and breaking tensile force

Type of joints	Form of presenting the equation	Obtained Equation	Coefficient of determination (R^2)
Heat-treated	Coded	$Y = 4022.32+347X_1-456X_2+1084X_3-340X_2X_3+1055.72X_1^2- 819.28X_2^2 – 849.28X_3^2$	0.85
	Real	$Y = -31290.94 - 748.41X_1+103954X_2+547.97X_3 – 226.66 X_2X_3+16X_1^2- 81928.27X_2^2 – 3.77X_3^2$	
Untreated	Coded	$Y = 4698.99 – 39X_1 - 575X_2+1071X_3$	0.45
	Real	$Y = 5052.98 – 4.87X_1-5750X_2+71.4X_3$	

Based on the sign of the coefficients of obtained models, it was found that the heat-treated wood joint strength increases when:

- the dowel length increases;
- the distance between holes ***increases;***
- the ratio of dowel penetration in the main part of joint decreases.

In the case of untreated wood joints was found that strength increases when:

- the dowel length increases;
- the distance between holes ***decreases;***
- the ratio of dowel penetration in the main part of joint decreases.

Based on the value of coefficients of models, it was found the most important independent variable that affects the strength of joint is the length of dowel. Moreover, the length of dowel has a nonlinear effect on breaking compression force that was obtained both for joints made of heat-treated and untreated wood (Figs.6a and 6b). In the case of breaking tensile force, the nonlinear effect of the length of dowel was observed only for joints made of heat-treated wood (Figs.6c and 6d).

In the case of heat-treated wood joints the independent variables interact for a better compressive and tensile strength, as it could be observed based on the equations presented in Tables 2 and 3. The most important interaction is between the ratio of dowel penetration in the main part of joint (X_2) and the length of the dowel (X_3).

Based on the developed models, the optimisation algorithm that is included in the Design-Expert Software and technological constraints, it was obtained a single optimal solution both for heat-treated and untreated wood joints. The solution implies to have a distance between dowels of 32 mm; a ratio of dowel penetration in the main part of 0.55 and a dowel length of 60 mm. The fulfilment of optimization criteria (D) was higher in the case of heat treated wood joints (D = 0.91) than in the case of untreated wood joints (D=0.74). The optimisation criteria consisted in maximizing both the compressive and tension breaking force.

The optimal values obtained are close to those found in the literature, as follows:

- the distance between dowels is suggested at 32 mm due to technological constraints (distance between axes of the drilling tools mandrels)(Cismaru 2009):
- the ratio of dowel penetration in the main part of joint is recommended to be 0.50 (Curtu et al. 1988, Craftmanspace 2016);
- the dowel length could be either 50mm or 60mm (Curtu et al. 1988, Negreanu 2003).

The selected optimum solutions were, also, experimentally verified. The sample size was of nine joints for each requested test. The values obtained for each sample are shown in Table 4. The relative error obtained for each model was calculated using equation (1).

$$\varepsilon = \frac{F_E - F_S}{F_E} 100 \quad [\%] \qquad (1)$$

where: F_E - is the compression or tensile breaking force applied to joint that was experimentally determined, in N;

F_S - is the compression or tensile breaking force applied to joint that was calculated with the developed mathematical models, in N.

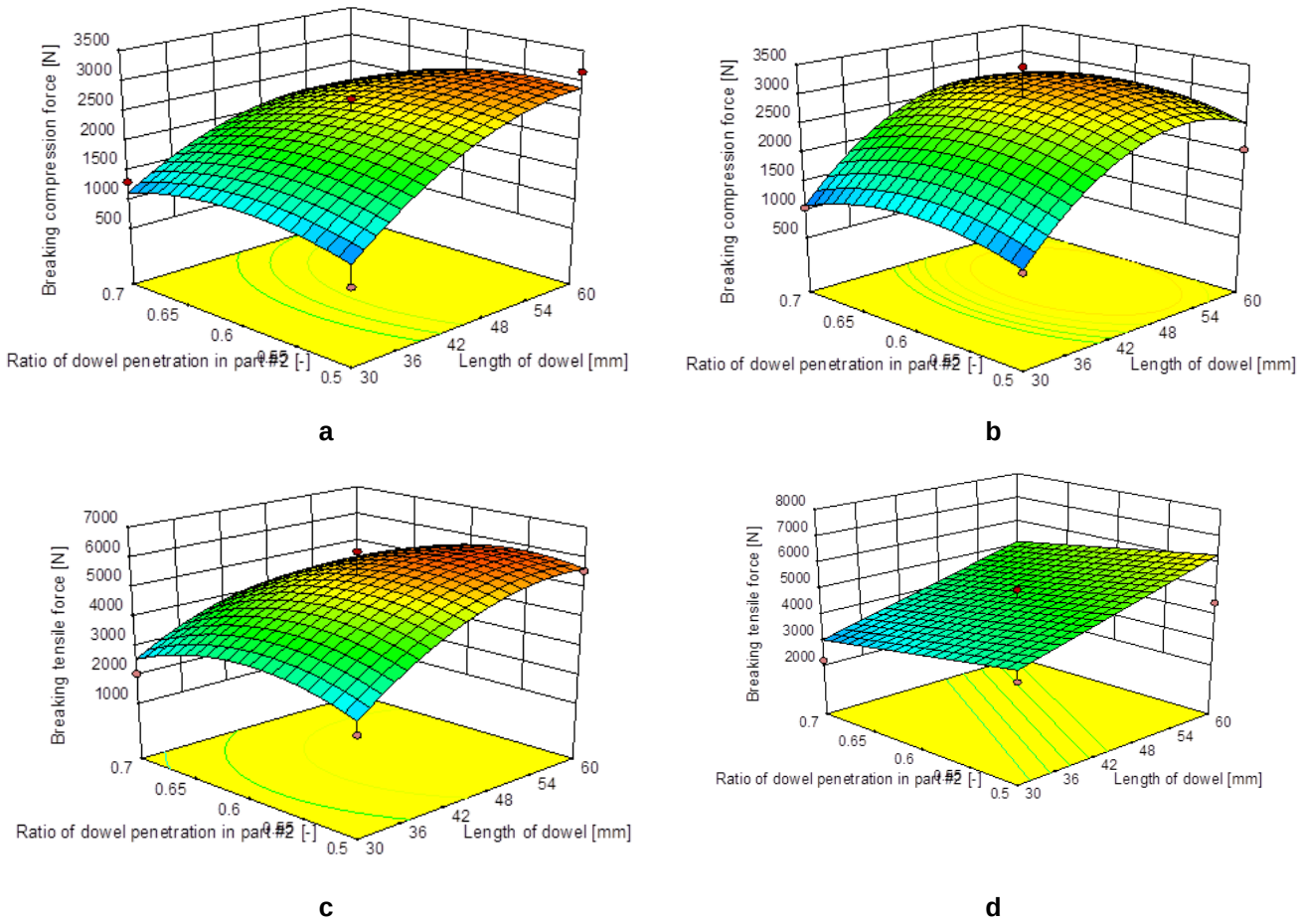


Fig. 6.
Response surface plot showing the effect of ratio of dowel penetration in the main part of joint and the length of dowel on the breaking compressive force (a - heat-treated material and b - untreated material) and breaking tensile force (c-heat-treated material and d – untreated material). The distance between dowels was considered equal to 32mm.

Table 4

The relative error obtained for the optimal solutions

Type of material	The method of determination	The distance between dowels, mm	The ratio of dowel penetration in the main part of joint	Length of dowels, mm	Compression breaking force, N	Tensile breaking force, N
Heat-treated wood	Mathematical model	32	0.55	60	2912	5900
	Experiment				2429	4779
	The relative error of modelling, in %				-20	-23
Untreated wood	Mathematical model	32	0.55	60	2901	5836
	Experiment				3205	5235
	The relative error of modelling, in %				9.5	11

CONCLUSIONS

The end to edge butt joint made of heat-treated wood have a breaking compression and tensile force lower than the joints made of non-treated wood. However, several heat-treated joints made an exception to that rule. Therefore, a study is under way in order to check the obtained results and to figure out this unexpected finding. Also, it was found that the compressive breaking force is generally smaller than the tensile breaking force both for heat-treated wood joints and untreated wood joints. The main variable affecting the resistance joints is the length of dowels. The obtained optimal solution could be considered suitable both for the joints made of heat-treated wood and to joints made of untreated wood. In a further study more variables that influence the compression and tensile strength of end to edge butt joints must be considered, in order to develop practical recommendations needed during the design phase of wooden products.

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COLOUR INFUENCE OF THE LASER RASTER SPEED ON WOOD PYROGRAPHY

Adrian PETRU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: adrianpetru_ro@yahoo.com

Abstract

*The paper presents the results of experimental research performed on sycamore maple wood (Acer pseudoplatanus L.), burned by a CO_2 laser. The influence of speed and lightness for CO_2 laser burning of sycamore maple wood have been determined. In order to evaluate the colour modifications, which may occur, the samples were treated at different speeds, the other work parameters were kept constant. To evaluate the aesthetic changes, CIEL*a*b* colour measurements were applied. Results showed that the lightness varies from 0.7 to 55.6. The interval limits of speed variation were defined from 75 to 500mm/s. Moreover, main observed effects near to interval limits are discussed. The wood had serious degradation, which increased lightness, at speeds under 75mm/s. No major lightness differences were observed at speeds near 500mm/s. The regression equation was defined. It was shown that the lightness depends on laser speed. Increasing the burning speed increased the lightness, too. The overall conclusion of this research is that the feed rate influences the lightness, but into limited intervals. These findings will be useful to be included in computerized databases for the automatic implementation of laser processes. An application of this study would be into the manufacturing of furniture and other products.*

Key words: *pyrography; wood burning; laser technology; colour; feed rate.*

INTRODUCTION

The colour is an important aesthetic characteristic of wood. Considering that, in our daily life, many objects are made from wood and they have purpose to increase our comfort. Pyrography is a method of increasing aesthetic properties, in the wood industry, by burning in a controlled manner. An attractive design increases sales and consumer satisfaction. Dyeing methods are usually adopted for traditional plain decoration, but these methods can initiate problems, such as chemical pollution and material coverage. Alternatively, laser pyrography can directly mark images on the wood, keeping the natural material texture and colour. The colour change of wood by pyrography is mainly due to changes in its chemical composition. The wood colour will become darker, if it is treated at high temperature. Thus, without any addition of chemical substances (as in the case of coatings), the aesthetical value of wood is increased. Petutschnigg et al. (2013) has used laser technology to increase the aesthetic value of skis.

Leone et al. (2008) observed that lasers are widely used in cutting and welding operations. Kincade and Anderson (2008) estimated that more than 40,000 cutting machines using CO_2 lasers have been installed worldwide. Unlike other plain decorating techniques, pyrography has some advantages, for example: it is an inexpensive technique, ecological, and available. Several advantages of using CO_2 laser irradiation on wood were observed by Kacík and Kubovsky (2011). In contrast to conventional colouration methods, lasers can change colour (from natural wood colour to black) only by delivering energy in the form of electromagnetic radiation.

The speed of the head along the X axis, named as the feed rate, is one of the important parameters in the engraving process because it influences the productivity. Theoretically, the feed rate can vary considerably, from 0 mm/s to supersonic values. A speed of 0mm/s is not used for engraving because the processed surface is very small. A surface given by the diameter of the laser spot is difficult to observe with the eyes. On the other hand, the operating principle of the equipment in raster, involves the movement of the working head at a speed no matter how small, but greater than 0mm/s. In practical terms, this range is limited by several factors:

- Technological possibilities – processing equipment;
- Processing possibilities – the physical phenomena that influences the processing;
- Productivity – Getting the desired effect in the shortest time.

Hernández-Castañeda et al. (2011) studied the influence that working laser parameters have on surface colour. They found that the traverse speed is the third most influential factor in the multiple-

pass laser cutting process of pine wood. It is directly related to the interaction time between the laser beam and the material, which increases or reduces the irradiance of energy in the cut process, especially when this factor interacts with laser power.

The feed rate influences the quality of the process. Processing is better at low speeds. At high feed speeds, the wood surface is improperly processed due to insufficient irradiation time. In view of the above, it is clear that the choice of feed rate is a real challenge, or the choice of working regime, because high speed increases productivity, but quality decreases, and low speed produces good quality but low yield. In their study of the influence of working parameters on quality laser processing, working for cutting stainless steel by pulsed Nd:YAG laser, Ghany and Newishy (2005) showed that the laser cutting quality depends mainly on the cutting speed, cutting mode, laser power and pulse frequency and focus position. Riveiro et al. (2010) performed cutting tests in pulsed mode. They demonstrated that the application of laser cutting techniques to process an aluminium–copper alloy can give a good-quality result, with adequate cutting rates. Also, Riveiro et al. (2010) indicated that high cutting speed and good quality can be obtained using high laser powers and focusing the laser beam onto the surface of the workpiece.

Comparing with the cut, the burning grade can be associated with the heat affected zone (HAZ) effect. Hamoudi (1997) showed that, during cutting by a 2 kW CO_2 laser assisted by 10 bar of nitrogen, increasing the cutting speed leads to narrow HAZ. Biermann et al. (1991) and Stournaras et al. (2009) have done some experimental work to explore the influence of HAZ parameters on the cut quality.

Usually pyrography is made on wood, but Irish (2012) proposed more materials used for pyrography, for example: leather, gourds, cloth, and paper. Researchers from the University of Warwick proposed MDF as a support material for laser pyrography (Howard 2014). The global trend in the use of lasers for fast growing wood species was observed by Petutschnigg et al. (2013).

OBJECTIVE

The aim of this work is to study the influence of feed speed on the darkening of the surface of sycamore wood. It has evaluated the colour in terms of luminance, or lightness, a component of the CIEL*a*b* system. The study proposed to obtain the variation law and range limits of the feed speed depending on lightness, without greater degradation of wood.

MATERIAL, METHOD, AND EQUIPMENT

The base material used in this study was sycamore maple (*Acer pseudoplatanus* L.) solid wood. The mechanical properties of this material are listed in Table 1. Cismaru and Cismaru (2007) recommend this species in the wood industry as: piano, violin and double bass parts; parquet and panelling; aesthetic veneers; chairs, table tops; and Filipovici (1965) appends to this list: turned and milled objects. This species is one of the most used species, recommended as a support for pyrography (Filipovici 1965, Bucur 1978, Walters 2005, Neill 2005, Easton 2010, Millis 2013, Gregory 2014). Sycamore maple is an important wooden material used in industry. Thanks to its aesthetic properties (colour and texture), it is suitable to be pyrographed by laser. Meier (2017) describes sycamore lumber as sapwood colour ranging from almost white, to a light golden or reddish brown, while the heartwood is a darker reddish brown. The white colour provides the possibility of obtaining a large colour gradient after burning. Grain is generally straight, but may be wavy. It has a fine, even texture. This texture provides the possibility to create a wide range of pyrographed patterns. Stanciu et al. (2015) observed that sycamore is spread all over the mountain and piedmont of the Carpathian area and in economic terms, the price of sycamore logs at timber auctions in Europe is somewhere around € 640/m^3 at the time of writing. Sycamore wood is much appreciated. Antonoaie et al. (2015), discussing the Brasov - Covasna area, showed that if there is at least 5% sycamore in the total wood volume, this is one of the factors that would persuade a manager to bid to a selling price 40% higher than the asking price at auction.

The wooden material used in the present research consisted of 235×85×10mm boards. In order to analyse the colour of wood, the tangential surface of samples was used. The work surface was chosen to contain mature and juvenile wood strips. The specimen boards were dried at 12% moisture content and conditioned at 20ºC temperature and 65% relative humidity as considered by Cismaru (2003). Before laser processing, the wood specimens were sanded with 80 grit sand paper and then sanded with 120 grit sand paper.

The equipment used was: Laser Engraving Machine 4030lsct, HP LaserJet 3055 all-in-one multifunctional printer for image scanning, PC for image processing, measurement and data analysis.

Table 1

Physical and mechanical properties of Sycamore Maple (Acer pseudoplatanus L.), according to Meier (2017)

Average Dried Weight	615 kg/m^3
Specific Gravity (Basic, 12% MC)	0.48, 0.62
Janka Hardness	4,680 N
Modulus of Rupture	98.1 MPa
Elastic Modulus	9.92 GPa
Crushing Strength	55.0 MPa
Shrinkage	Radial: 4.5%, Tangential: 7.8%, Volumetric: 12.3%, T/R Ratio: 1.7

Because the literature does not provide clear information in this way, it is necessary to study a wide range of speed variation, from 500 to 0mm/s. The experiments were performed with a 40W CO_2 slab laser. In order to study the variation in feed rate for 16.5W laser power, 20 experiments were designed. Feed rate variation ranged from 25 to 500mm/s, with steps of 25mm/s between experiments. Of these, 19 experiments were made because at the feed rate of 50mm/s the wood ignited. This phenomenon is not acceptable for pyrography. For this reason, no experiments were performed at values lower than this feed rate. The other operating parameters were kept constant. The experiments conducted in pulsed mode were performed varying one parameter (just feed rate). The ranges of engraving parameters are summarized in Table 2. For each working regime a square surface measuring 20mm was processed. In order to compare the results, the experiments were performed in the direction of the wood grain.

Table 2

The range of variation of the engraving parameters

Processing parameter	**Value**
Laser power (W)	16.5
Feed rate (mm/s)	50 … 500
Pulse frequency (Hz)	20000
Scanning gap (mm)	0.0254
Focal length (mm)	73
Focus position	Surface
Assist gas	Compressed air
Nozzle diameter (mm)	5
Stand-off (mm)	30

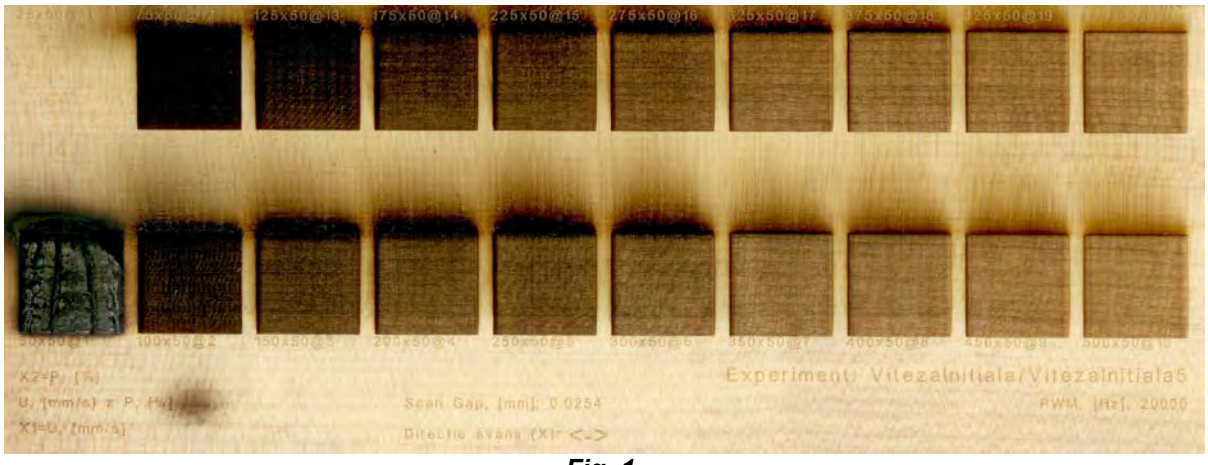

Fig. 1.

Colour scale produced of sycamore showing the effect of speed on wood colour.

The experimental plan was transposed into the machine program using a specialized laser equipment program. The equipment had been adjusted before work was started. A resulting sample is shown in Fig. 1. As can be seen, the arrangement of the experiments was achieved in two rows parallel to the wood grain. Using this arrangement, a number of errors relating to the inhomogeneity of the wood, such as: the annual ring width, the proportion of late and early wood, etc., have been avoided. Drawbacks relating to material preparation such as the cutting direction, fibre direction, etc., have been avoided, as well. Mainly, all these influence the colour of the wood surface. Between processed surfaces, unprocessed areas were also left in order to have the reference surfaces as close as possible to the processed ones.

In order to measure colour, the processed samples were scanned with an HP LaserJet 3055 all-in-one scanner. The selected parameters for this study were:

- Resolution: 600 dpi;
- File format: bitmap;
- Colour mode;
- Scan scale 1:1.

These were set in order to analyse the scanned image file that was transferred to a computer.

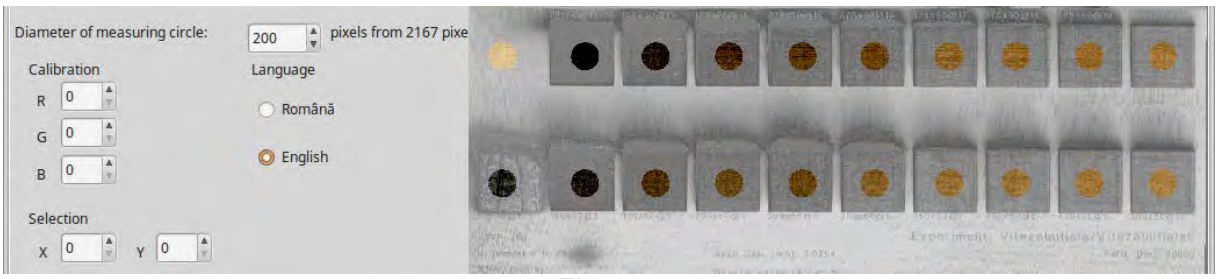

Fig. 2.
The template with measuring points.

The colour was measured using the method presented by Petru and Lunguleasa (2014). Inside of each filled square, the colour was measured at 200 pixels diameter for a circular surface. It resulted in 31587 pixels for each square and 631771 measured pixels for the whole experiment. To simplify all measurements, an electronic template was made using all the measured pixels. Each area, reference and pyrographed, was measured using this template (Fig. 2). To evaluate the colour changes, CIEL*a*b* (1976) colour measurements were applied. This measurement system consists of three coordinates, named: *L**, *a**, and *b**. The *L** coordinate represents lightness and it is on a scale of 100, where L*=100 is white and L*=0 is black. The *a** coordinate characterises the green (negative values), and red (positive values). The *b** coordinate characterises the blue (negative values), and yellow (positive values). This method was preferred because it expresses the colour directly through the lightness parameter, which represents the degree of darkness of the processed surface, respectively the degree of processing. For each measured round area, the average value was calculated for each colour coordinate. The differences between these values, its dispersions and colour change were calculated by using equations (1). The minimum and maximum values were also found.

The colour change was calculated for each colour coordinate (*L*, a** and *b**) as related to its reference area and each processed area. Colour differences between the reference and processed surfaces were calculated by using the following equation as defined in (BS EN ISO 105-J03:1997):

$$\Delta E^*_{ab} = \sqrt{\left(\Delta L^*\right)^2 + \left(\Delta a^*\right)^2 + \left(\Delta b^*\right)^2} \tag{1}$$

where:

$$\Delta L^* = L^*_P - L^*_R$$
$$\Delta a^* = a^*_P - a^*_R$$
$$\Delta b^* = b^*_P - b^*_R$$

P= Pyrographed sample
R= Reference sample

The measured values were recorded and for each speed the average, minimum, maximum, the most often encountered, deviation and standard deviation of colour were calculated. The regression equation was determined.

RESULTS AND DISCUSSION

The trichromatic colour parameter variations, measured in the CIEL*a*b* system, are shown in Fig. 3. The variations of the three colour components are different:

- The *L** parameter, which represents the lightness, has the highest variation and increases proportionally to the increase in the feed rate;
- The ***a**** parameter, which represents the green colour for negative values and red colour for positive values, has the smallest variation of the trichromatic parameters. Note that this parameter has a negative value at the feed rate of 50mm/s, but at this rate the sample ignited, therefore the result is not included in the present study;
- The b* parameter, which represents the blue colour for negative values and yellow colour for positive values, has a variation smaller than the L* and greater than the a* parameter but closer to the latter. The variation of this parameter can be divided into two distinct areas:
  - At speeds less than 225mm/s, where the values are below the reference value;
  - At speeds greater than 225mm/s, where the values are above the reference value.

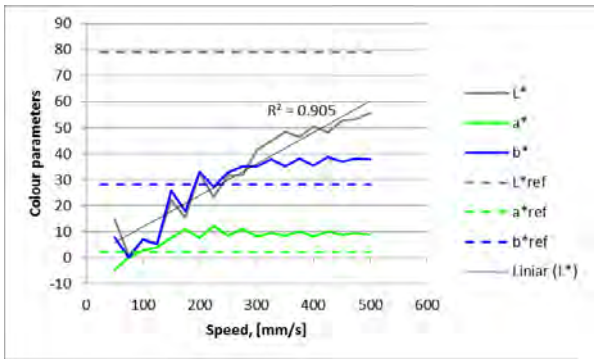

Fig. 3
Average colour variations based on head speed.

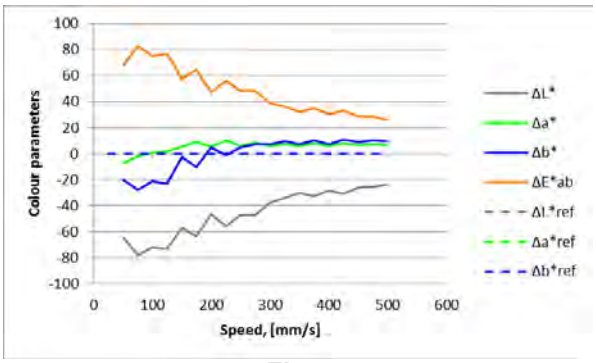

Fig. 4
Colour changes depending on speed variation. (Please note that the reference samples are all set at zero).

The trichromatic colour differences between the reference surface and the processed surfaces, measured in the CIEL*a*b* system, are shown in Fig. 4. As can be observed, the most influential component, which affected colour difference ΔE_{ab}^* is ΔL^*, it has the greatest variation and the curves of these two parameters are similar. ΔL^* varies directly with the increase in speed, respectively, if the feed rate increases, the surface will be lighter. The other two components, Δa^* and Δb^* have a small influence on colour change, compared to the ΔL^*. By comparing treated/processed to reference areas, the first thing one can notice is that the treated areas display the tendency of getting darker (negative values of ΔL^*). Therefore it can be concluded that the simplified analysis of differences in colour nuances need only consider the L^* factor. The darkening tendency of the laser pyrographed samples is stronger during speeds of under 250mm/s.

At first glance, the variance trend of the L^* parameter seems to be linear. The R^2 factor is 0.905. It means that the trend accuracy is very good for an inhomogeneous material such as wood. The tendency is mathematically correct, but physically it is incorrect because, as shown above, processing at the very low feed head speed (0mm/s) is no longer possible, and at very high speeds the wood surface does not change its color because the irradiation time is insufficient to transfer enough energy to the wood surface.

Also in Fig. 3 it can be noticed that there are insignificant changes in colour at high feed speeds. These differences are highlighted by comparing the colour with that of the reference surface. The reference lightness is not equal to 100 because the natural colour of the wood is neither white or uniform. At feed speeds lower than 150 mm/s a decrease in lightness is observed. This decrease cannot be continuous because the speed cannot be 0mm/s. It is advisable to avoid low speeds for the following reasons:

- Low productivity;
- Low speed causes heating and attrition of the head moving mechanism;

- Studying the effect of speed and processing gas on laser cutting of steel using a 2 kW CO_2 laser, Hamoudi (1997) observed that little dross formed at low speeds of less than than 1 m/min and the best cutting quality was achieved at 2 m/min. In the case of wood this phenomenon does not carbonise the inorganic components, which increases the surface lightness.

In Fig. 3 it can be noticed that the variation of the *L** component has three distinct intervals of variation:

1. At feed rate values of 50mm/s or less, the wood is burning. The results obtained in these cases were excluded from this study.
2. At an advanced speed of less than 125mm/s, the lightness is almost constant. Working modes with lower speeds than this value are not recommended because there is no greater blackening of the surface. Using speeds in this range generates an unjustified increase in working time.
3. At an advanced speed of higher than 125mm/s the lightness increases. Variations of lightness can be likened to an exponential variation. This is the speed range to be considered further.

Also, in Fig. 3 it is observed that the lightness at high speed (with the minimum surface energy), of 500mm/s, is quite far from the natural colour of the wood. For that it is recommended to use powers less than 16.5W. On the other hand, in Fig. 3 it is shown that the lightness at low speed (maximum surface energy), at feed rates around 75 mm/s, is 0.74. This value is quite near to a black colour (the lightness of 0). In the same figure, it is shown that the lightness at high speed (minimum surface energy), at feed rates around 500 mm/s, is 55.59. This value is quite far from the reference colour (the lightness for natural wood colour), which is 78.97. It is recommended to use powers lower than 16.5W.

When investigating CO_2 laser cutting of 2024-T3 alloy, Riveiro et al. (2010) following a mathematical modeling, observed that the size of the heat-affected zone at cutting (that is similar to the blackening of pyrography) is influenced by the cutting speed. They believe that this is probably a consequence of the energy released in each processing condition. Modelling the colour lightness variation according to the shape of the exponential function is preferable, because its boundary limits the range of the speed function by setting the upper limit of the speed variation of the laser head, which influences the colour of the treated wood. Considering the above, namely defining an equation that would accurately express the physical phenomena of changing the colour of wood by varying the speed of the laser head, it was proposed to analyse the experimental data. This experimental data was analysed after a predefined function. The trend of lightness variation is exponential. The law of variation is given by the equation:

$$L^*(u) = 73.67 - 95.02 \; e^{(-u/295.81)} \tag{2}$$

where:

- *e* is the basis of the natural logarithm (Euler's number);
- *u* represents the feed rate, in mm/s;
- *L** represents the lightness;

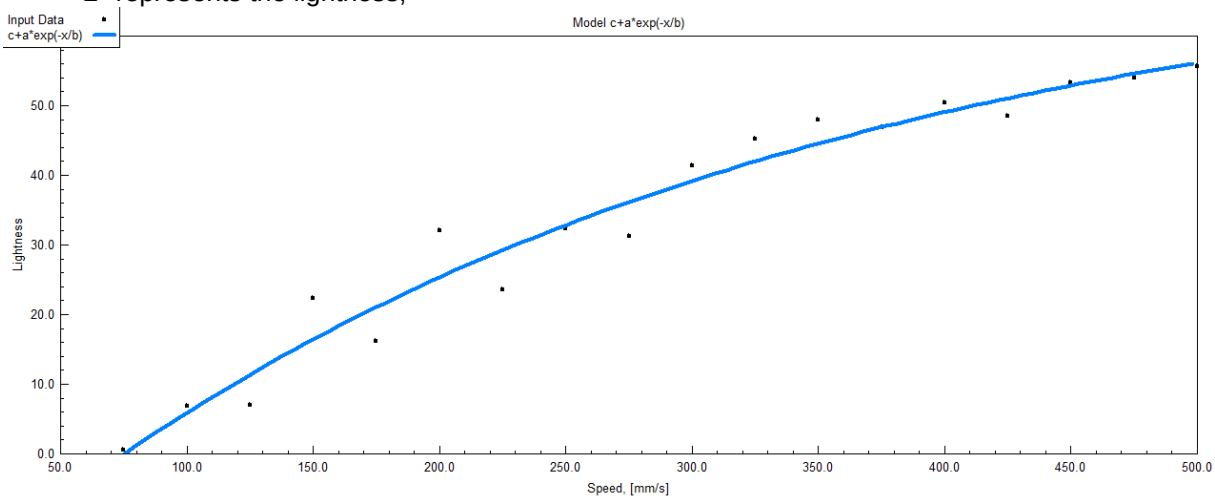

Fig. 5
Lightness variation curve according to speed.

Coefficient of Multiple Determination (R^2) is 0.96, which means that the approximation of experimental results with this variation law is very good, especially for a non-homogeneous material such as wood. The lightness variation curve was designed using equation (2). The lightness variation curve according to speed is shown in Fig. 5.

CONCLUSIONS

The obtained results within the present research demonstrated that the speed has an important influence on wood colouration by laser, even when the other parameters are kept constant. Both early and late sycamore wood have a good colouration using this technology. However, the variation range is limited by physics phenomena happening during laser processing.

Three lightness variation ranges corresponding to the feed rate were identified and defined. Of these, only one interval fulfils the necessary conditions to obtain different luminances used in pyrography. This range from 75 to 475mm/s was defined.

This study confirms the theoretical suppositions, which considers that the lightness variation corresponding to the feed rate is exponential, because processing at the very low end of the feed head speed (0mm/s) is no longer possible, and at high speeds the wood surface does not change its colour because the irradiation time is insufficient to transfer its energy to the wood surface.

At speeds less than 75mm/s there is a large amount of degradation of the material, which shows an increase in lightness. This means that obtained lightness at speeds less than 75mm/s can also be obtained at higher speeds. From this observation it follows that the use of feed rates of less than 75mm/s is not recommended because it is not productive.

The determination of the maximum feed speed is conditioned by the possibilities of the machining of the equipment as well as by the experimental observations that at higher speeds there is no significant change of colour. At feed rates higher than 475mm/s an increase in lightness is noted. This increase is due to the insufficient irradiation time, and the processing cannot be achieved. The same effect can be obtained by using lower laser power. Due to shortcomings in low productivity at low speeds, it is recommended that the variation of the other factors are also studied. Considering the differences between the wood species, the results obtained are valid only for sycamore wood. The method of work can also form the basis of research for other woody species.

Lightness regression equation has been defined according to the feed rate for the studied power. Using this equation, the lightness can also be determined for other working parameters. The laser equipment has been chosen correctly because the range of variation is within the processing possibilities. A detailed analysis of the influence of laser feed rate on the wood was made. The structure of the support material may influence the processing, therefore, the average mean deviation of the measured values has been determined for each measured surface.

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SECTION 8. WOOD CONSTRUCTIONS

LONG SPAN PORTAL FRAME MADE FROM GLULAM AND CLT MOUNTED TOGETHER USING ONLY CARPENTRY JOINTS

Tõnis TEPPAND
Estonian University of Life Sciences, Institute of Forestry and Rural Engineering,
Department of Rural Building
Fr. R. Kreutzwaldi 5, 51014 Tartu, Estonia
Mob: +3725045693, tel +3727313184, E-mail: tonis.teppand@emu.ee

Abstract

The paper presents the results of concept design for modelling, designing and producing long span portal frames and constructing buildings with them.

Ordinary portal frames of steel structure are not suitable in many cases for a variety of reasons. They are heavy and very fragile to transport, are not fire resistant, are present special difficulties in use because of possible corrosion and are always not satisfactory architecturally. Wood-steel assemblies such as flitch beams are also complicated because of possible condensed water on metal and fasteners.

Constructions using GLULAM and CLT (especially in the last decade) show rising trend. Timber has very many good properties as a building material if we know or remind ourselves of the skills how to handle it. The biggest enemy of wood and the reason for decay is moisture. All structures and finishing materials need very careful and qualified design to avoid collection of water on surfaces and consequent increasing moisture content.

This paper describes a method appropriate for the 21st century of how to design portal frames of GLULAM and CLT panels and construct of them without metallic fasteners.

Key words: *portal frame; hardwood pins; stiff eave node; carpentry joints.*

INTRODUCTION

Portal frames are commonly used for buildings that need long spans without any supporting system such as columns, load carrying walls or other systems similar to the column-truss system. Portal frames do not need any stiffening members perpendicularly such as diagonals or diaphragm walls that could spoil usage of the internal open area of the building. The perpendicular stiffness is assured with a structural scheme of only three hinge joints together with a stiff eaves joint. This enables them to be used in swimming pools, sport halls, riding grounds, buildings with inside active driving etc. The indoor climate must be controlled by heating and humidity control. Steel structures especially are vulnerable to condensation on metal parts that have the cold bridges through the insulated perimeter or in the zones above the dew point. It is preferable to use timber or timber-based structures in such types of buildings and even better without metallic plates and fasteners at the connection nodes. GLULAM and CLT technologies based on polyurethane glue do not have the decay problems in wood if other technical solutions support it.

Carpentry joints can succeed today with rapid development of modelling and designing software supported by CNC-technologies. A lot of old and known carpentry joints are used and the new ones come into being every day.

The present design gathers together different kinds of modelling and is based on several experiments on structural elements of wood and/or of timber-based materials in the Estonian University of Life Sciences who have given us additional knowledge and helped us to understand the already forgotten skills of carpenters from the past (Seliste and Teppand 2011).

OBJECTIVE

The main objective of the present concept design was to find out the technical solutions of how to use GLULAM and CLT in most optimised way to produce portal frame details with three hinged joints and to avoid using ordinary metallic fasteners as much as it is possible. All negative know-how was taken into consideration to avoid the problems they cause. All the positive know-how from the different tests and skills of old and present time was used in this concept design.

MATERIALS AND METHODS FOR PORTAL FRAMES

Comparison of materials

Steel portal frames are heavier than GLULAM and CLT. Because steel frames require transport and mounting (need bigger cranes), they cost more. Steel frames have less stability in exposure to fire. It is very difficult to avoid cold bridges if the structure must be continuous through the external insulated perimeter. Emission of CO_2 is one of the biggest among building materials. GLULAM is the most commonly used for beams or beams/rafters and it has often been used for portal frames too. Metal plates and lot of pins/bolts have been used in the eave node connection to make it stiff.

CLT-panels are currently used for walls.

Is it possible to use these technologies together to produce a new type of portal timber frames without metallic fasteners that are easy to mount at the building site?

Moisture in the wood

The problem of timber structures in the open environment is occurrence of condensation on metallic parts because of temperature drop on cold nights. This phenomenon wets the timber around the metallic fasteners. Slow decay can develop in the timber around the fasteners and therefore cause the joint to slacken after which the geometry of the whole structure can change and values of inside forces in it. Is it possible to avoid it at all or decrease the problem?

Fire protection

The other important difference is behaviour materials in fire. Steel does not burn but loses its stability at temperatures higher than 600°C when it becomes plastic and may collapse. Load carrying structures of steel as frames need to be protected against fire. Fire protecting coatings may only last 5 years and only some of them keep their fire resistance properties until 10 years. After that the old coating must be removed and needs to be changed. It is technically very complicated and expensive work. The quality at the building site is not the same as done in the factory.

There are very high requirements for the surface quality for undercoating before the fire protection coating is applied. Normally it needs to be shining metal but many surfaces of the steel profiles cannot cleaned of old coating because of too small gaps between the details. How to get the result following fire regulations and enough fire resistance time?

RESULTS AND DISCUSSION

Modelling, designing, structural analyses and fire-resistance

To design a portal frame the software Rhinoceros was used that has enough functionality and is even the best for modelling curved and/or two-curved surfaces. It is able to convert the modelling fail-format to the STEP-format using it with FEM-software (Finite Element Modelling) to make structural analysis. After finding the correct dimensions for cross-sections, a new type of carpentry joint at the eave node CNC machining format was generated for CNC-workstations.

During the designing process it is necessary to calculate the char speed of timber elements in the fire which in GLULAM and CLT is normally 0,65mm/per min. An increase in the size of cross-section of the elements of timber to get the needed fire-resistance time is allowed. This helps to avoid fire protection coatings that may not last the whole life-span of the building.

Construction materials and production method

The portal timber frame with a stiff eaves node has parts where it can use the best properties of both GLULAM and CLT.

The beam/rafter is made as GLULAM. The lower end of it at the eaves node has a routed dovetail male joint (Fig. 1). The upper end has a Knuckle Joint Hinge to connect the two different beams/rafters at the ridge node (Fig. 2).

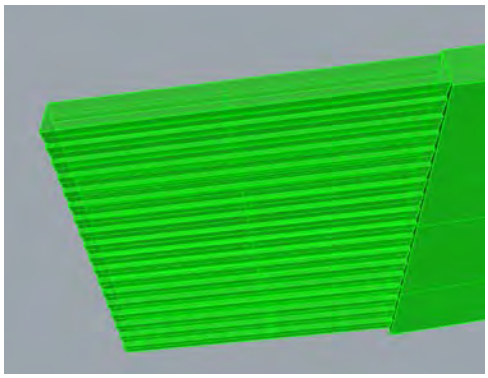

Fig. 1.
Dovetail male joint of the beam/rafter at eaves node.

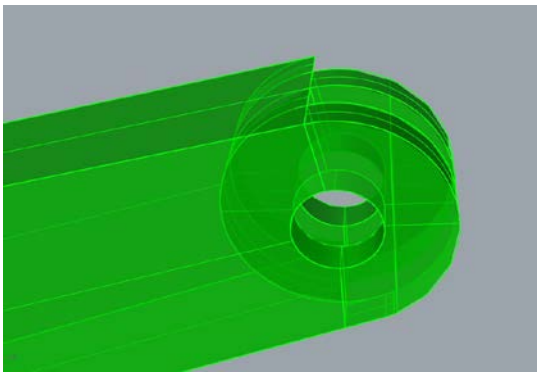

Fig. 2.
Knuckle Joint Hinge to connect two different beams/rafters at ridge node.

The post is made of two CLT-panels to get the horizontal stiffness and load carrying to a maximum value vertically at the same time. Two panels were used because of the need to rout dovetail female joints for the beam/rafter into the upper (Fig. 3) and mortise (Fig. 4) to the lower part of the post. Two parts of the post will be mounted together with beam/rafter between them at the building site.

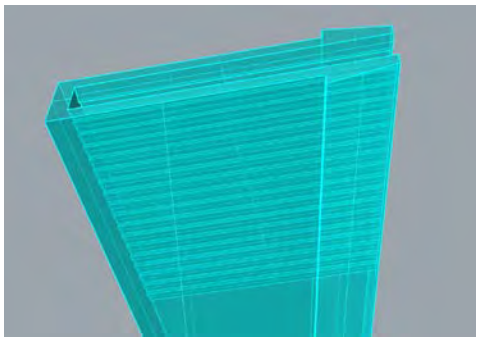

Fig. 3.
Dovetail female joint of the beam/rafter at eaves node.

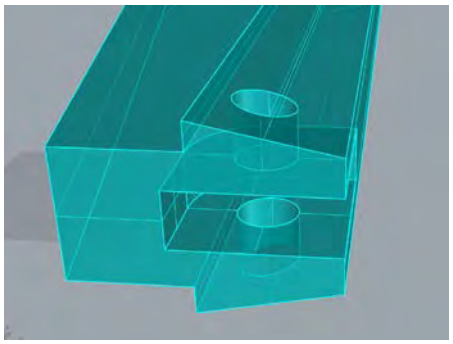

Fig. 4.
Mortise on the lower part of the post at base node.

Hardwood pins were used to fix the different members together as in the past (Fig. 5). The pins do not carry over the loads. If the structure consists of softwood members (mostly of spruce) and pins of hardwood (mostly of oak) the speed of drying shrinkage and moistures expansion is different which keeps the connections tight.

Fig. 5.
Pin of hardwood.

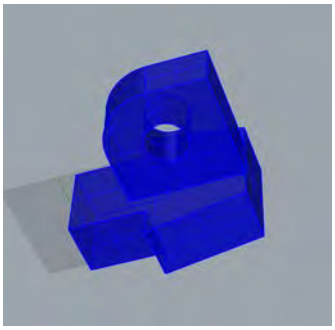

Fig. 6.
Support of hardwood.

Special supporting elements (Fig. 6) of hardwood (mostly of oak) are used under the posts to protect them. These elements are easily changeable if decay occurs and prevents moisture damage to the posts.

Mounting technology

The portal frame can be mounted on the building site with the help of two mobile cranes.

Firstly the posts will be mounted comprising two half pieces with beam/rafter together (Fig. 7). The barbed pins or pin-screws of hardwood are used to connect them. Both parts of the frame lay horizontally. The hardwood supports are fixed to the foundation after that. Then both sides of the portal frame can be lifted onto the supports. The connections are then fixed with hardwood pins. Last of all the ridge connection is fixed with a hardwood pin (Fig. 8).

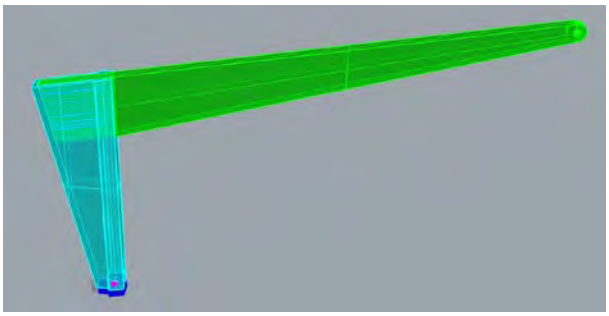

Fig. 7.
One half of the portal frame.

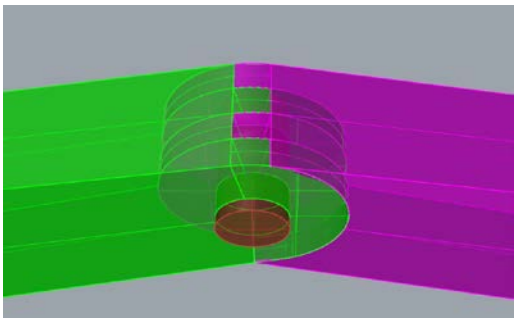

Fig. 8.
The ridge node fixed with a hardwood pin.

CONCLUSIONS

Using GLULAM and/or CLT in structures for portal frames needs careful handling because of different details needed to assemble them on the building site. It is most important to ensure the same moisture content in wood until all the details have been mounted in place so as to avoid damage and decay of timber elements because of moisture. Decay in timber can be minimised if the connections does not use metallic fasteners. The next important thing is geometry. The correct geometry of the whole structure from general to the smallest detail needs to be considered. Success with carpentry joints together with the development of CNC-workstation functionality permits the design and production of complicated joints even without gluing. The old skills with hardwood pins can be used to make corrosion and decay free connections between the members that need to act as hinge joints. Scheduled maintenance works are very important to get the desired lifespan of structures. They must be part of the project design developed by the architect and structural engineer. It is not possible and would be very expensive to predict every last detail for the total lifespan, thus designers have to predict/calculate the realistic lifespan of them. If some of the elements do not last until the end of total lifespan - such as supporting details under the posts - they have to design for easy (cheap) replacement technology.

ACKNOWLEDGEMENT

The patent application submitted for designing and producing technology of portal frames of GLULAM and CLT without metallic fasteners.

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RESEARCH UPON CREATING A SYSTEM FOR CONVERSION THE WATER KINETIC ENERGY OF HILL AND MOUNTAIN RIVERS IN GREEN ENERGY, USING RECYCLING MATERIALS

Virgil GRECU
Transilvania University of Braşov, Faculty of Wood Engineering
Universităţii Street nr. 1, 500068 Braşov, Romania
Tel: +40 0729924092, E-mail: vgrecu@unitbv.ro

Abstract

In the present paper is presented a system for generation of green energy which uses the power of streams water flow and is situated in mountain and hill areas. The hydro electric system consist of: a turbine which is in fact a wheel with wooden cups, a spindle to transmit the rotation motion, wheels for transmission of rotation, accessories, subsystem to transform the mechanical energy in electrical energy, unit for electricity storage, subsystem for collecting and transport of energy.

Recycling materials were used for electrical system: wood, steel, subassemblies resulted from agricultural machines or other installations out of order, or resulted from the Centre for Recycling Materials.

The whole system was designed to be rapidly and economically manufactured, with a minimum technical endowments.

An optimum management system is aimed to distribute the water flow rate from streams or derivates, from the main riverbed, so that the biodiversity and specificity of the local area will be not affected.

Key words: *wooden installation; hydro energy; green energy; Romanian traditions.*

INTRODUCTION

The present paper is a part of an extensive study entitled "Research for the settlement of new technical solutions of electrical power supply for Romanian traditional constructions from the Fagaras Mountains area, in the context of promotion of sustainable development programs."

The system presented in this paper promotes some solutions to regularly produce the electricity by means that can be easily found in the adjacent farms, small production units, holiday houses. As well, it could be an alternative for energy production in the case of calamities, when the energy from standard sources will be not available. (Grecu 2015)

The last but not the least, the promotion for non-invasive utilisation of mountain stream courses, to protect the environment, taking over the stream flow by derivation for short distances of maximum 500m and return to the stream, was considered. This fact will contribute to maintaining the biodiversity of waters throughout the year, without the risk of dry out when the rainfall is reduced or absent. (Grecu 2015)

OBJECTIVES

Four objectives to carry out this research were proposed:

1. To identify Fagaras County as location of the system, the place where Romanian traditional water-mill machines worked in the past ;
2. Designing a new system for generation of green energy using the hydro power of hill and mountain streams;
3. Elaboration of an optimal management system for distribution of water flow rate to not disturb the biodiversity and the local communities;
4. The execution of the energetic system using recycling materials from the industrial and agrarian constructions, or from the Centre for Recycling Materials.

MATERIALS, METHOD, EQUIPMENTS

Preponderantly **recycled materials** were used for the proposed energetic system which is, in fact, a mini hydroelectric plant for conversion of water kinetic energy into electrical energy.

The barrage (Fig. 1) was performed from spruce logs processed on two surfaces. They derived from trees with knots and other defects, rejected from industrial processing. The diameter of the trees was approximately of 250mm and the length 2300mm.The logs were horizontally super

positioned and finally embedded at the ends in concrete structure (4). As seen in (Fig. 1) and (Fig 2) the structure of the barrage had a squared shape to give strength and protection for electrical and mechanical equipments. A squared structure ensures the resistance and secure of the turbine spindle.

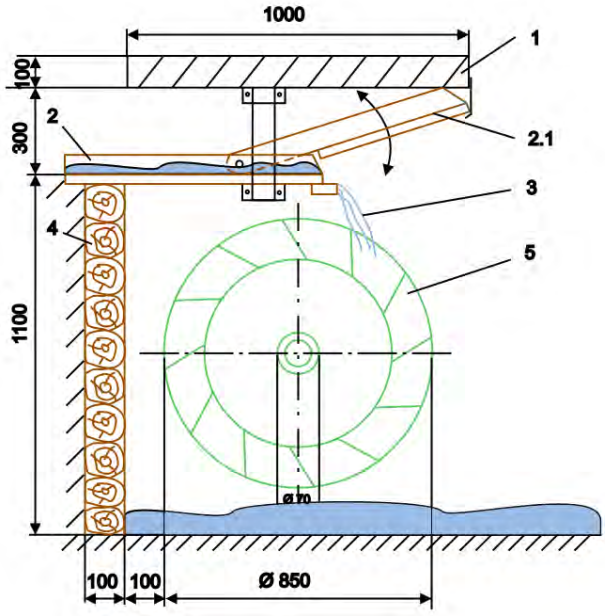

Fig.1.
Cross section through the barrage, turbine, wooden spouts and bridge sustaining the mobile spout- Principial scheme
1 – bridge sustaining the mobile spout; 2 – fast spout; 2.1 –mobile spout 3- water flow rate; 4 – logs barrage; 5 – turbine.

In the concrete structure were included sand and rocks extracted from the streambed (Fig 2).

Turbine was manufactured using a spindle from an agricultural machine, with a diameter of 70mm and a length of 2300mm. Two steel covers from a barrel were fixed on it at a distance of 800mm. 12 wooden cups between covers were fixed with screws. The wooden elements of cups originated from reused concrete formworks (Fig 2).

The fast and mobile spouts were manufactured from the same wooden material.

The equipments consisting in belt wheels, intermediary spindle, were reused from agricultural equipments out of order, from a local farmer.

The alternator used for variant 1 for electricity generation and accumulator for energy storage originated from Dacia automobile.

The barrage was built on the previous emplacement of the old barrage that supplied with water the mill machine, using logs and soil as waterproofing materials. The energo-intenssive (cement) materials have been strictly limited to the protection and safety area of the construction.

Designing of the barrage, construction reinforcement and function of the turbine and equipments envisaged the topography of the land and the management of the water flow.

Thus, the barrage was elevated to distribute a quantity of water on an adjacent canal towards the old barrage, to ensure an independent "staircase" course for maintaining the aquatic biodiversity and migration of the fish fauna.

Before construction of gutters and canal for bringing the water into the pool, a mapping of the water stream was done. The main purpose of it was to irrigate the agricultural lands.

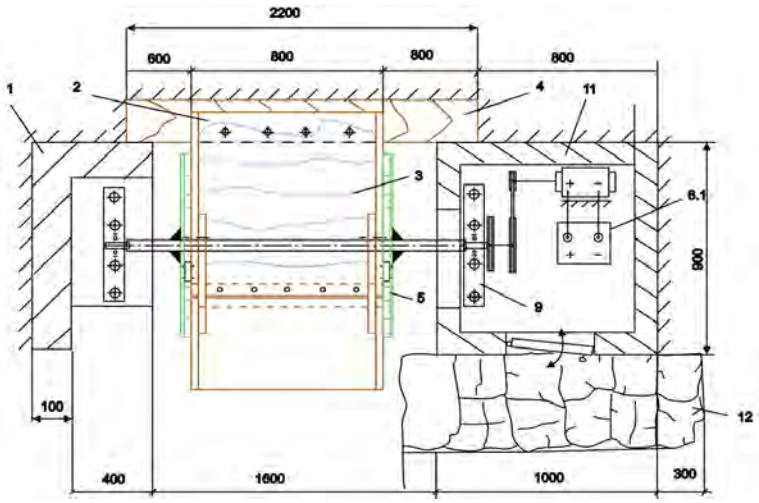

Fig.2.
Longitudinal section through the concrete structure, turbine and wooden spouts – Principial scheme - Variant 2 including a generator unit
1 –edged concrete structure; 2 –fast/ immobile wooden spout 3 – water flow rate; 4 – log barrage; 5 –mobile spout; 6 – generator unit ; 6.1 –consumer; 9 - bearings; 11 – concrete structure.

RESULTS AND DISSCUSION

First objective of identifying the location for hydro electric system was reached. It was Fagaras Mountain area, specific for development of handicraft techniques from ancient times. The land of emplacement was chosen on a stream derived from the main river, that have almost a constant water flow in time and that cross an agricultural land.

Designing the small hydro electric station started with location and then the execution of subsystems and electric components.

The main components are presented in (Fig. 1), (Fig. 2) and (Fig. 3). The system is considering two variants: the first variant V1 proposed that electricity is generated by an alternator and for the second variant V2- the electricity is generated by an electric three-phase generator.

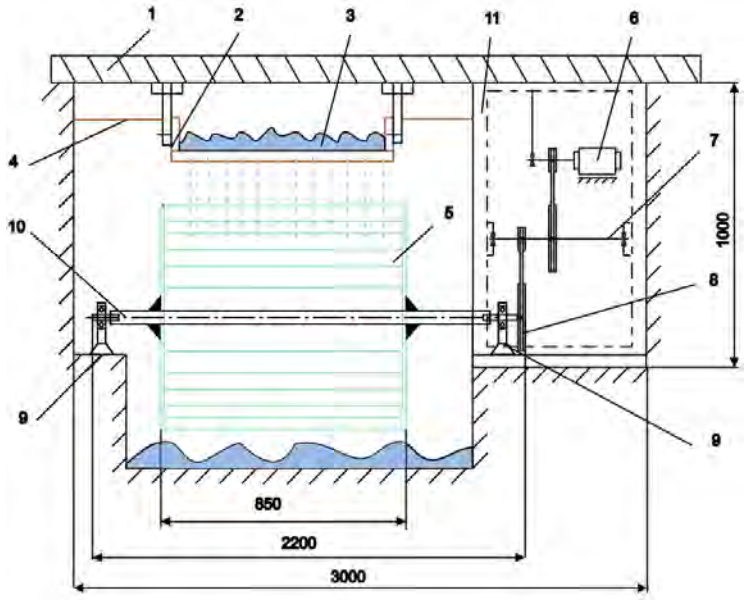

Fig.3.
Longitudinal section through the bridge- Principial schema
Variant 1- with alternator
1 – overpass; 2 – fast spout;3 – water flow rate;4 – wooden barrage; 5 - turbine; 6 – alternator; 7 – trapezoidal transmission belts; 8 – pulley; 9 – spindle guide bearings; 10 – turbine spindle; concrete structure.

A kinematic chain with intermediary spindle to transmit the rotating motion upwards of 1500rot/min was used for the **Variant 1.**

The transmission ratio is not constant; it depends of water flow rate and transmission loading based on slip coefficient.

The efficiency of belt transmission has a maximum value when the loading is optimum and consequently at an optimum slip coefficient.

The slip coefficient for trapezoidal transmission belts (k) is 0.02 (Manolescu 1998).

The calculation formula for the ratio of transmission motion (Radu 1997) according to the slip coefficient (k) is :

$$I = \frac{n_1}{n_2} = \frac{d_2}{d_1(1-k)} \qquad (1)$$

where: I = transmission motion
n1 = rotation speed of the turbine spindle (rot/min)
n2 = rotation speed of the secondary spindle (rot/min)
d1 = diameter of the wheel on turbine spindle
d2 = diameter of the wheel on the intermediary spindle
k = coefficient of trapezoidal belt.

The main spindle (n1) generates 65-70 rot/min according to the variation of the water flow rate, approximately 30 l/s.

If the diameters d1 and d2 are known, n2 could be calculated using the formula (1) as:

$$n_2 = \frac{n_1 \cdot d_1 \cdot (1-k)}{d_2} = 343 \text{ rot/min} \qquad (2)$$

where: n1 = 70 rot/min
d1 = 400 mm
d2 = 80 mm
k = 0,02

The rotation speed of the alternator spindle (n3) was calculated with formula:

$$n_3 = \frac{n_2 \cdot d_3 \cdot (1-k)}{d_4} = 1680 \text{ rot/min} \qquad (3)$$

where: n2 = 343 rot/min
d3 = 400 mm (diameter of the third wheel on the intermediary spindle))
d4 = 80 mm (diameter of the wheel on the alternator spindle)
k = 0,02

After calculation, the rotation speed of the alternator spindle (n3) was 1680 rot/min. It allows the whole system to function as an electric system with a battery of 12 volts similar with an automobile electric system.

The diagram for generation, conversion and utilisation of electric energy is presented in Fig.4.

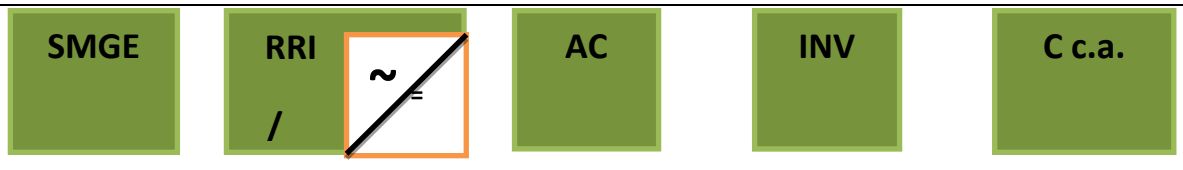

Fig. 4.
Block diagram of making a hydroelectric system of energy generation (Grecu 2015)
a - Mechanical generation system; b - rectifier and charging regulator; c - Battery; d - Inverter; e – Consumer.

Variant 2 aim to use a three-phase generator and manufacturing a short kinematic chain: wheel on the main axe of the turbine – transmission wheel on the generator axe .

The rotation speed was calculated with formula (1) as:

$$n_2 = \frac{n_1 \cdot d_1 \cdot (1-k)}{d_2} = 343 \text{ rot/min} \qquad (4)$$

In care: n1 = 70 rot/min (rotation speed of the turbine)
d1 = 400 mm (diameter of the turbine spindle
d2 = 80 mm (diameter of the hydro generator spindle)
k = 0,02

For a rotation speed 340 rot/min, the generated electricity could be used for different activities (e.g. lighting).

For the present research a higher rotation speed was not tested. The mini hydro electric dam station described above is presented in (Fig. 5).

a **b**

Fig.5.
Mini hydroelectric dam station
a – Front view– stream derivation; b – Right view– mini hydroelectric dam station.

In Fig.6 are presented in detail the main components of the hydroelectric station.

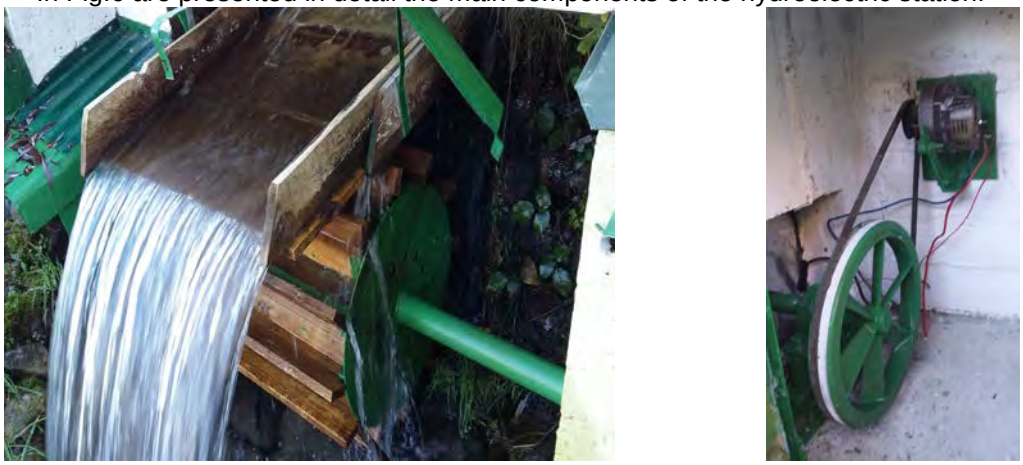

Fig.6.
Mini hydroelectric dam station
Detail of component parts: wooden fast spout and mobile spout, turbine with wooden elements and spindle.

The third objective envisaged the optimal management system for distribution of water flow rate (Fig 7). As a result of the optimum management of the water flow rate, electric power is produced and environment protection was achieved as main activities. An additional facility to irrigate the surrounding agricultural land when draught was obtained.

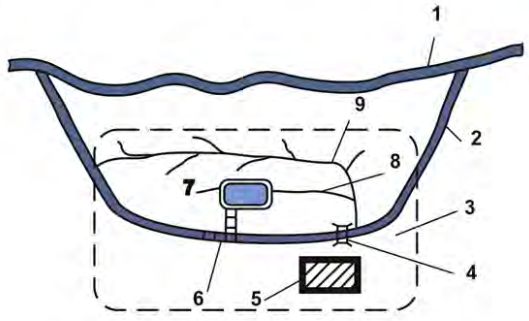

Fig.7.
The management system for distribution of water flow rate
1 –main stream (streambed); 2 – derivation of the stream; 3 – agricultural land; 4 – mini hydroelectric station; 5 – mini construction; 6 – stairs for water flowing from pool; 7 – pool for aquatic fauna regeneration; 8 – penstock channel; 9 – irrigation canal.

The old pool for watermill machine and the penstock channel were rehabilitated by underpinning with river stones. As consequence, the biodiversity was maintained and improved. The water flows through a staircase wooden spouts. The proposed system allows to the free circulation of the aquatic fauna on the secondary course of the stream and the possibility of its regeneration into the pool (Fig. 8).

a **b**

Fig. 8
Distribution of water flow rate
a – left view- deviation of the stream and water pool; b – right view – pool and mini hydroelectric station.

The execution of a traditional mini hydro electric system using recycling materials available in agrarian households and additional new equipment, by allocation of low investments has a big potential, in the context of sustainable development.

In (Fig. 9) and (Fig. 10) is presented the hydropower system.

Fig. 9.
Detail for turbine and equipment Turbine ongoing – The mobile spout is up.

Fig. 10.
Detail for turbine and equipment- Front view- Turbine at rest.

CONCLUSIONS

The presented hydro electric system based on ecological principles without a major intervention in natural environment and using traditional and recycling materials offers the following advantages:

- Reveals the Romanian traditions and rebuilds the specificity of the Fagaras County in terms of handicraft techniques.
- The system construction allows building adjacent constructions with a specific architecture and design such as "local fingerprint".
- The old irrigation channels for agricultural lands and orchards will be activated.
- The management of water flow rate will lead to optimal distribution of water quantity to produce electricity and to increase the agricultural production.
- A large amount of recycling material as subsystems could be used for a long period of time.
- As previously presented in similar studies (Grecu 2015) the mini hydropower dam station is an integrated system of electrical "green energy" production. The project includes additional two systems for generation of electricity by wind force and solar radiation. Thus, when one of the agents that generate energy is not available its functions can be fulfilled by the other one.
- The construction of the station described above on a short distance of 500 m, is an alternative to the present proposed system of mini hydro power stations, more expensive and non-friendly with environment.
- The aim of this project to produce "green energy" was to use the local materials, on the existing streams or derivates, so that minimum electricity could be obtained even in times of stress, when life of people is in danger.
- The project is ongoing and will continue with designing of other new subsystems of electrical power generation by using the wind force and solar radiation, as well as a fourth system by using the geothermal energy.

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DETERMINING THE BEHAVIOR OF THE BASKETBALL, ON VERTICAL DIRECTION, ON SURFACES OF FLOORINGS CONCEIVED FOR GYMNASIUMS

Olimpia-Minerva ȚURCAȘ (DIACONU)
Transilvania University of Brașov, Wood Engineering College
Str. Universității nr.1, 500036 Brașov, Romania
E-mail: minerva2013diaconu@gmail.com

Adriana FOTIN
Transilvania University of Brasov, Wood Engineering College
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: adrianafotin@yahoo.com@*unitbv.ro*

Ivan CISMARU
Transilvania University of Brașov, Wood Engineering College
Str. Universității nr.1, 500036 Brașov, Romania
E-mail: icismaru@unitbv.ro

Abstract

This work presents the results of the experiments run on a mosaic flooring (equated to concrete) and a beech wood flooring model created by us for gymnasiums (A-type panel), according to SR EN 12235, with the help of the sport flooring testing device conceived and manufactured with this purpose within Transilvania University of Brașov, called ***D-DTPS-3****.*

This endeavor is part of the PhD thesis bearing the title ***"The influence of structure upon the mechanical properties and energetic absorption of parquet-type sport hall wooden floors".***

The goal is testing fewer flooring stuctures conceived and manufactured by ourselves using local kinds of wood for gymnasiums and see if they meet the requirements and exigencies regulating this type of floorings.

Key words: *wooden floorings; sport floorings; rebound; ball; testing device.*

INTRODUCTION

Currently, the situation of wooden floorings is dynamic, concerns and research in this field trying to provide improvements/optimizations to the specific properties of wood. Due to its qualities, wood has chances to keep on being a successful material (Cismaru 2006, Cismaru et al. 2015).

A special category of wooden floorings is represented by floorings dedicated to spaces for practising dance, ballet, aerobic gymnastics and any form of exercise or sport. They are considered *special* because of their "*behavior*" as response to the activity that takes place on these floorings. (Cismaru et al. 2015).

Specialists in floorings are concerned with finding that optimum balance between flexibility and rigidity, so that the user should not make an extra effort but not suffer injuries or accidents either, as well as obtaining flat surfaces – by reducing the unlevelings of the support layer (Pardoseli Magazin 2013).

Hence the great variety of floorings for sports that underwent a considerable development, mainly in what concerns the support on which the wooden panels rest on, from the most traditional system to the most innovative one (depending on possibilities and performance levels wanted for sport activities).

Despite the appearance of new materials, wooden flooring systems for sports such as basketball, squash, and dance, remain invincible because the elasticity must occur on an extended surface, not on a single point, therefore the wood is preferred over rubber (*kineticsport.ro*, "*Products*" section, "*Gymnasiums*" category, position: "*Wooden Sport Surface*", 9.11.15/h 13.41).

Testing as a scientific process in the world of sports and recreative games needs a highly specialised expertise (Kolitzus 2012). Over the last decade, many research centers developed in this field, among which stands out ***Del Tec*** from Holland (*deltecequipment.com*), developer and manufacturer of such special (mobile) sport flooring testing equipment.

Subsequently to the study run on 51 different flooring materials (Demker 2009), the elaboration by Swedish Standards Institute of a global standard for measuring the mechanic comfort for all flooring types, both in situ and in the laboratory, was recommended, the testing methods existing in EN 14808

and EN 14809. As in this field, at international academic level, published studies and research results are very rare, we are constraint to refer only to those mentioned above and the regulations imposed by standards and established sport institutions (*European & International Standards. Surfaces for sports areas. A short guide. 2014*).

OBJECTIVE

The objective of this research is realising floorings with a nationally high grade performance, competitive with the ones previously mentioned. We will watch the graph of the rebound of the basketball on a concrete/mosaic surface that will be considered as a yardstick in comparing the response that the wooden structures proposed by us will give. By means of this method we verify if the structures conceived and manufactured meet the requirements imposed by sport institutions as well as by national and international standards for gymnasiums.

MATERIAL, METHOD, EQUIPMENT

In order to design correctly the wooden floors dedicated to this type of activity – sports – preliminary testings were performed consisting of bending testing samples made of steamed domestic beech wood of different thicknesses (15, 20, 25, 30mm), widths (30, 40, 50, 60mm) and lengths (300, 350, 400, 450mm) to observe the behavior and make possible a right choice regarding the dimensioning of the friezes for the panels that will be tested. Following the preliminary analysis, it was chosen for the parquet friezes to be 20x50x500mm. The friezes from the carpet were arranged/assembled according to the English model.

The parquet friezes were manufactured in the Multi-functional Wood-processing Workshop (HI 5) within the Wood Engineering College of Transilvania University of Brasov, going through the following stages (Fig. 1):

a) severing – circular saw

b) splitting – circular saw

c) face-edge straightening – straightening machine

d) planing to width – width-planing machine

e) calibrating – wide tape sanding machine

f) cutting to length - circular saw

g). groove milling – normal/vertical axis milling machine (MNF); 5 mm mill

h). groove milling – normal/vertical axis milling machine (MNF); 5 mm mill

Fig. 1.
Realising parquet friezes – Stages.

After manufacturing the parquet friezes, because the SR EN 12235 standard provides that the minimum area for testing must be at least 1,00m x 1,00m, the structures conceived have this area except the last one which is 1,50m x 1,50m. The friezes in the carpet were glued with JOWACOLL® 103.05, leaning on resinous wood beamlets. Fewer structure variants were realised, i.e. type A and type B panels leaning on 3 beamlets, the difference between them consisting of the dimension and

arrangement mode of the friezes. For panel C we have the leaning on 5 beamlets. For type D panels we have leaning on beamlets and traverses, the difference consisting of the 15, 20 și 25mm frieze widths. The last type E panel is 1,50m x1,50m and rests on beamlets and traverses.

Experimental research belonging to this work refer only to the type A panel structure. Its characteristics are presented in table 1 and type A panel structure is presented in Fig. 2.

Table 1

Type A panel - Characteristics

Structure type A panel (1,00mx1,00m)							
friezes				**beamlets**			
thickness	width	length	total	thickness	width	length	
mm	mm	mm	pcs	mm	mm	mm	pcs
20	50	500	40	20	40	1000	3

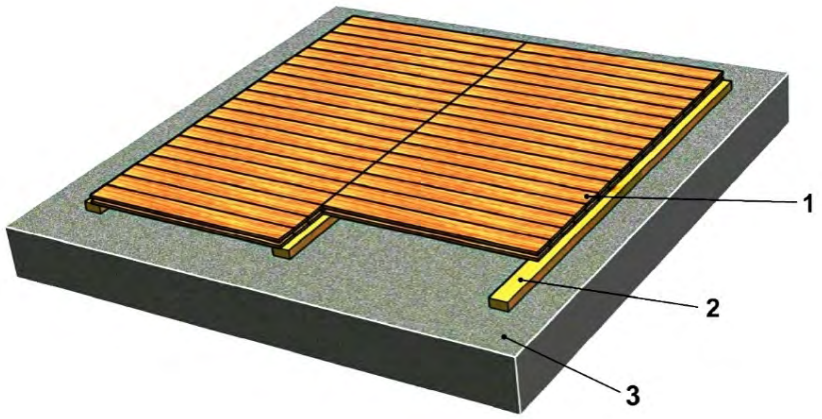

Fig. 2.
Type A Panel Structure
1 – beech wood parquet frieze; 2 – resinous wood beamlets; 3 – concrete support layer.

Beech wood parquet friezes, dimensions: 500x50x20mm are arranged like floors and glued with JOWACOLL® 103.05, resting on 1000x40x20mm resinous wood beamlets, disposed at a 500 mm distance between each other axis, fixed with 3,5x30mm wood screws (Fig. 2). The panel is attached to the support layer with 6x65mm screws.

The support layer for the tested structures/panels is composed of the mosaic flooring equated to the reinforced concrete layer over which quick primer for non-absorbing supports (Super primer from BAUMIT) was applied and a 2-3mm auto-leveling screed (Nivello Duo from BAUMIT) was cast to ensure flatness.

As working method, we used a procedure elaborated according to SR EN 12235 stipulating that the height of the ball rebound from a sport surface is calculated using the ecuation (1):

$$R\% = \frac{R_s}{R_c} \times 100 \quad (1)$$

where:
R% - the relative height of the rebound, as a percentage;
Rs - the height of the rebound from a sport surface, in meters;
Rc - the height of the rebound from concrete, in meters.

Therefore, we conceived a testing device meant to be in accordance with the provisions of SR EN 12235 standard, as well as the 2016-2017 General regulations for organizing basketball competitions (RGOCB).

The testing equipment presented in fig. 3 is conceived and realised within the premises of Wood Engineering College.

The functioning principle of the device consists of fastening the ball (RGOCB) inside a ring-band with 3 elements (2 fixed and one with mechanical retraction) and releasing it from a 1.80m height (acc. to SR EN 12235). By means of an ultrasound device – *Einstein*™ ***Distance Sensor*** (*Distance Sensor*

DT020-1) we measure the height to which the ball rebounds after falling on the panel subjected to the testing. The calculation of the rebound height is done using the ***MiLAB*** software installed on a dedicated digital tablet that allows data collection, display, and analysis, transforming the ball motions in relevant graphic representations.

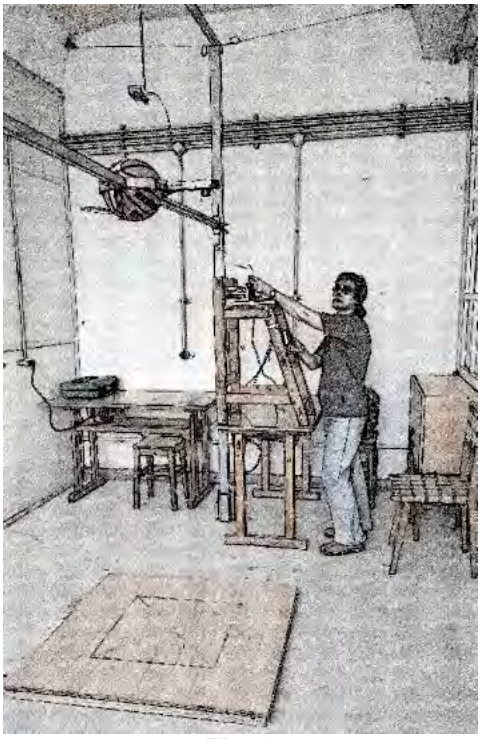

Fig. 3.
D-DTPS-3 Testing device.

The ball used for testings is the MOLTEN 7 ball, Fig. 4, the official game ball for competitions organized under the aegis of FRB, in accordance with the RGOCB.

Fig. 4.
Basketball used for testings.

RESULTS AND DISCUSSION

Within the experiments regarding measuring the value of the maximum rebound of the basketball on a concrete/mosaic surface, 5 (five) measurements were performed, according to the SR EN 12235 standard stipulating that the ball must fall from a 1.8m height.

Fig. 5 presents the variation of the maximum rebound corresponding to the five measurements for three diffferent inner ball pressures.

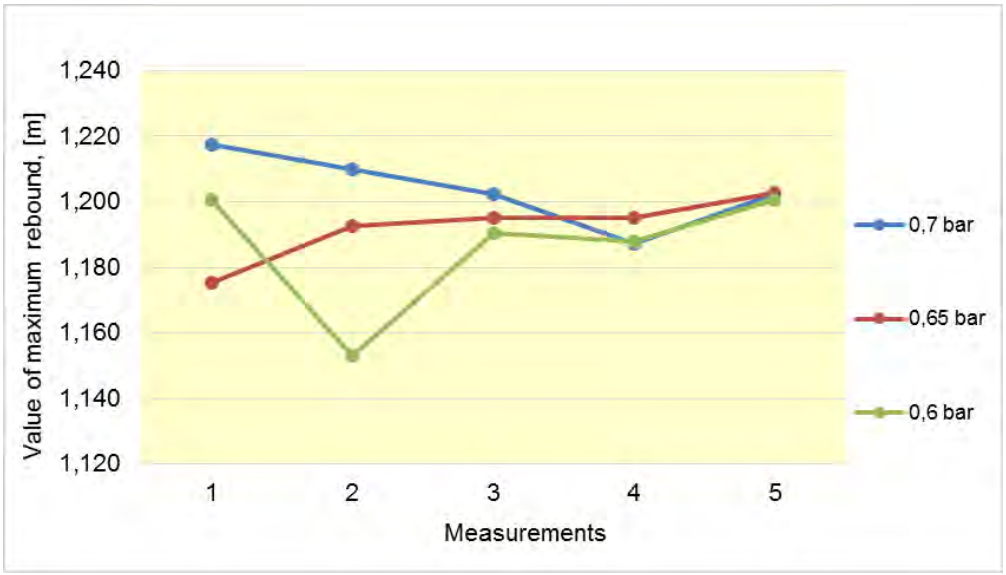

Fig. 5.
Variation of the maximum ball rebound on the concrete/mosaic surface at three different inner ball pressures.

Fig. 6 represents the maximum rebound of the ball inflated at three different pressures, related to the average of the five measurements, compared to the value stipulated in the standard.

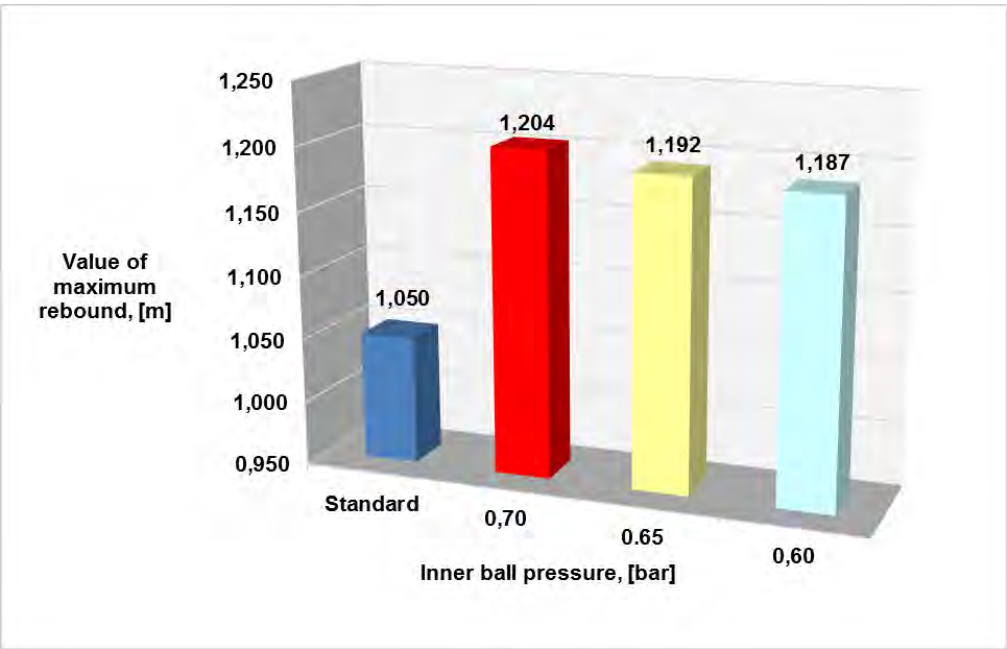

Fig. 6.
Comparative graph regarding the standard value and average of measurements of maximum ball rebound on the concrete/mosaic surface at three different inner ball pressures.

Thus, we find that the diferrent inner ball pressure, respectively 0.7, 0.65, and 0.6 bar influences visibly the basketball rebound when it dropped from a 1.8m height (acc. to SR EN 12235).

The graph below, Fig. 7, presents the results obtained by testing in 5 points a type A wooden panel.

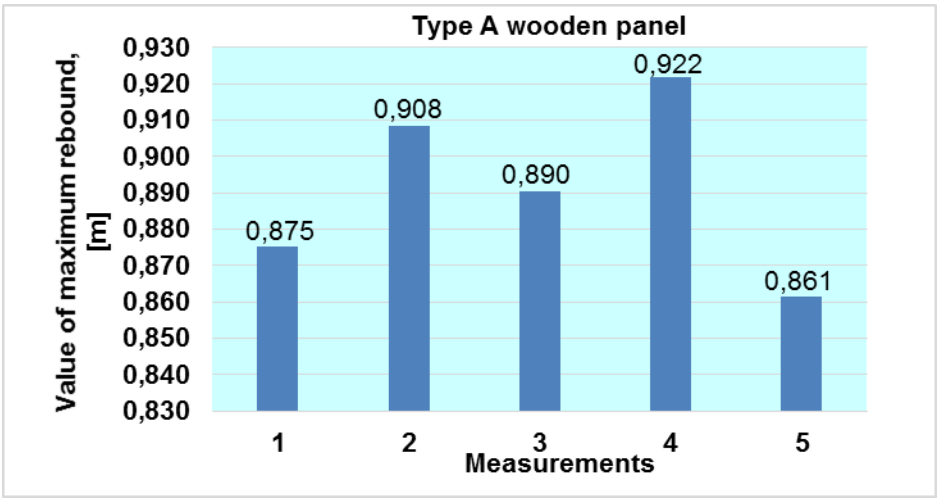

Fig. 7.
Graph for 0.7 bar inner basketball pressure using a type A wooden panel.

For sport floorings, the vertical aspect of the ball (after the impact with the floor), according to EN 12235 (basketball game), must be at least 90%.

Type A flooring made it to only 74%. This demonstrates that this type of flooring is not reccomended for halls where such sport activities unfold.

CONCLUSIONS

The results obtained from the testings performed on the concrete/mosaic surface demonstrate that the inner pressure of the ball used for testing has a very high influence as well. Therefore, we will do all testings on the structure conceived and manufactured in order to realise the PhD thesis with one inner ball pressure only, i.e. the 0.7 bar one. This way, the yardstick for concrete/mosaic will be 1.204m, all relations being done to this value.

After the testings performed on the type A wooden panel that has the structure composed of 20x50x500 mm friezes arranged as floorings and resting on 3 (three) beamlets, we found that this one responded with only 74% versus 90% which is the minimum accepted for sport floorings. Therefore, this type of flooring is no recommended for halls where basketball is played.

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SECTION 9.

COMPUTER-AIDED ENGINEERING IN WOOD INDUSTRY

THE USE OF CT-SCANNING TECHNOLOGY IN WOOD VALUE-CHAIN RESEARCH AND IN WOOD INDUSTRY - A STATE OF THE ART

Magnus FREDRIKSSON
Luleå University of Technology, Wood Science and Engineering
Forskargatan 1, 93187 Skellefteå, Sweden
E-mail: magnus.1.fredriksson@ltu.se

Olof BROMAN
Luleå University of Technology, Wood Science and Engineering
Forskargatan 1, 93187 Skellefteå, Sweden
E-mail: olof.broman@ltu.se

Dick SANDBERG
Luleå University of Technology, Wood Science and Engineering
Forskargatan 1, 93187 Skellefteå, Sweden
E-mail: dick.sandberg@ltu.se

Abstract

X-ray computed tomography (CT) is a powerful tool for the non-destructive measurement of dynamic processes in wood. After more than 25 years of research at Luleå University of Technology in the field of CT-scanning of wood material, the first industrial CT-scanners are now installed in sawmill production for the in-situ measurement of internal log features to steer of the sawmill process with the help of this information.

This paper provides an overview of the potential of CT-scanning in wood-material research and how this data can be used for the modelling and simulation of the wood value chain. A database of CT-images of trees is used to create a log model including the outer shape of the logs and their internal knot structure. Simulation software is used to saw these virtual logs in different positions relative to the sawblade, and also for the crosscutting of the sawn timber to components. The output is dimensions and grades of sawn timber, volume yield as well as an economic result based on real economic conditions. A specially designed climate chamber for CT studies of the drying of sawn timber is used to increase the knowledge of how the drying affects the response from the sawn timber during seasoning.

Key words: *CT scanning; sawmilling; simulation; modelling; drying.*

INTRODUCTION

X-ray computed tomography (CT) was introduced in the medical field in the early 1970s and uses X-rays to determine the density profile through a human body. Funt and Bryant (1987) were probably among the first to use medical CT images in the detection of internal log defects. They developed an automatic method for the interpretation of CT images to identify knots, rot, and cracks occurring in a log, but they also met the same problem as others at that time – the scanning time was about three minutes for a single 1 cm long disc or slice of the log. The technology was far from being realistic for industrial conditions. For more than 25 years, medical CT has been used in wood research at Luleå University of Technology (LTU). In wood science, CT is mostly used for steady-state studies of internal anatomical features of the wood material, but at LTU it is also possible to study processes such as drying, modification, water absorption, internal and external cracking, and material deformation in a temperature- and humidity-controlled environment (Fig. 1). This paper focuses however, on how CT-based information from logs (roundwood) can be used for simulation studies of the sawn timber value chain, both in research and for industrial purposes.

Computer simulation is an appropriate tool for studying the wood-value chain, for several reasons. Firstly, the complexity of both the raw material and the process itself means that it is diffcult to assess the possible effects of different decisions without a numerical model. Secondly, since the process of sawing and other machining operations is irreversible, the same material cannot be tested several times in a real system. This is possible however using a computer model, where the same log or board can be processed several times in different ways to study how different process parameters affect the outcome. Using a computer model, the effect of the raw material can be virtually neglected since only process parameters are changed between different tests. The opposite can also be done,

even though this is also possible in a physical test. Time is also an important factor. In industrial tests, the lead time between the start and end of a test is quite long, and in the meantime the production units are occupied by the testing. This is avoided in simulation studies.

To realize a truly integrated approach to the wood-value chain, it is important to be able to predict the final result of a decision taken early in the chain. Preferably, this prediction should be made before a saw blade has even touched the log. To do this, there must be a way to link the properties of trees to the properties of end-products. Such an approach is presented in this paper. A short introduction to the X-ray CT principles is followed by a description of the different tools necessary for the modelling and simulation of the early stages in the wood value chain, i.e. (1) a 3D-description of properties in roundwood – the virtual stem bank, (2) a simulation program for the sawing of the virtual logs in the stem bank, and (3) a simulation program for the cross-cutting of sawn timber. Finally, progress in the development of industrial applications based on CT technology will be presented.

OBJECTIVE

The purpose of this paper is to give a comprehensive overview of the possibilities of using CT-scanning in wood-material research and of how these data can be used for the modelling and simulation of the wood value chain for industrial purposes.

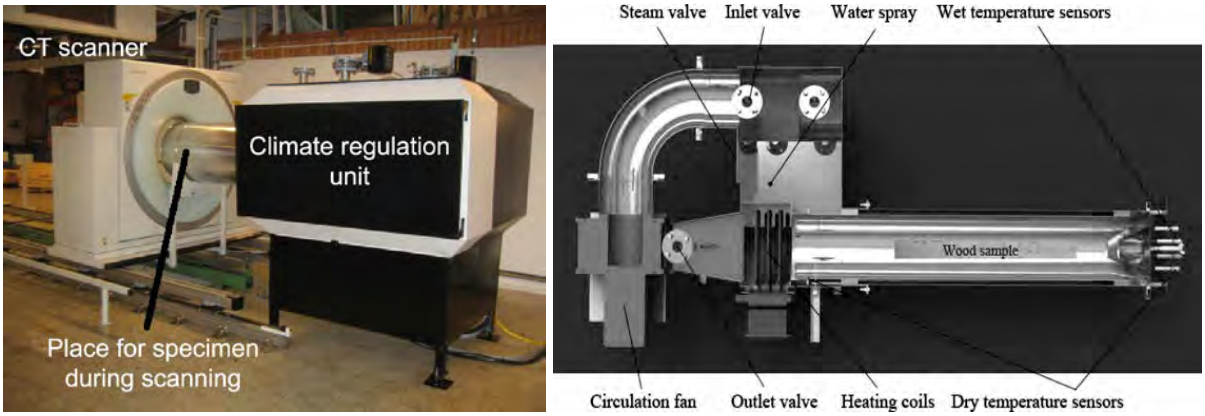

Fig. 1.
Siemens Somatom Emotion medical CT-scanner equipment at LTU with an integrated climate chamber for the non-destructive monitoring of dynamic processes within the wood material (left). Exploded drawing of the climate chamber (right).

X-RAY COMPUTER TOMOGRAPHY (CT-SCANNING)

X-ray scanners follow the theorem of Radon (1917) who theoretically demonstrated that the internal structures of an object can be reconstructed from single or multiple projections of the object, depending on the number of directions considered. In 1979, Cormack and Hounsfield received the Nobel Prize in medicine for the development of CT technology.

Whatever the number of directions, X-ray beams are sent and detectors measure the X-ray radiation transmitted through an object (Fig. 2). The intensity of the transmitted X-ray radiation can be related to the attenuation of the X-ray by the object by the Lambert–Beer exponential law under the assumption that the radiation is monochromatic and that the beam is propagated linearly in the object (Davis and Wells 1992):

$$I = I_0 e^{-\mu d} \qquad (1)$$

where: I is the intensity of the transmitted X-ray beam, I_0 is the intensity of the incident X-ray beam, d is the thickness of the object (length unit), and μ is the linear attenuation coefficient of the material along the transmission path (length unit^{-1}).

X-rays are emitted during rotation of the source around the object. At the same time detectors measure how many photons that have passed through the object. After complete rotation, the cross section is reconstructed showing a map of the attenuation coefficients. In medical CT-scanners, these values are normalized and compared to the coefficient of water, nowadays called the CT-number (Fig. 3).

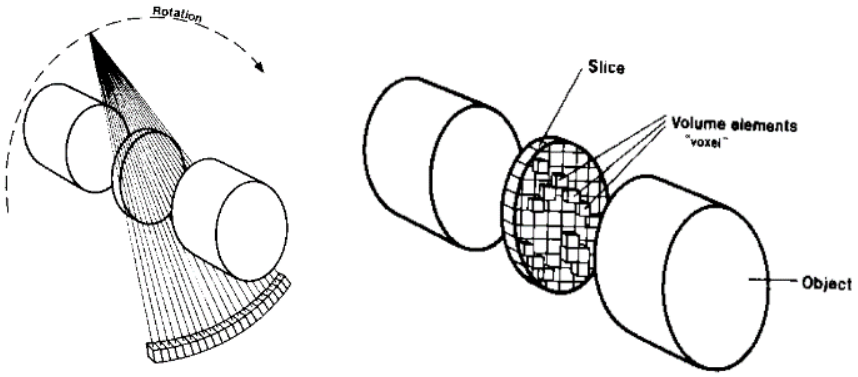

Fig. 2.
X-rays emitted during rotation are being detected and then calculated to give a map of attenuation coefficients.

In the model presented by Tsai and Cho (1976), the linear attenuation coefficient μ is the sum of two absorption coefficients, each of them directly proportional to the material density. Several other authors (e.g. Davis and Wells 1992; Lindgren 1992) have suggested that the linear attenuation coefficient μ can be directly related to the material density. The other factors involved are the chemical composition of the material (Tsai and Cho 1976) and the incident beam energy that is related to the type of source used. These factors are not independent. The material density ρ is the main influence with high-energy X-ray beams, whereas the chemical composition has its main effect with low energy radiation (Macedo et al. 2002). The relationship between density and attenuation coefficient for green (wet) wood is close to linear with an accuracy of the order of ±6 kg/m^3 (Fig. 3).

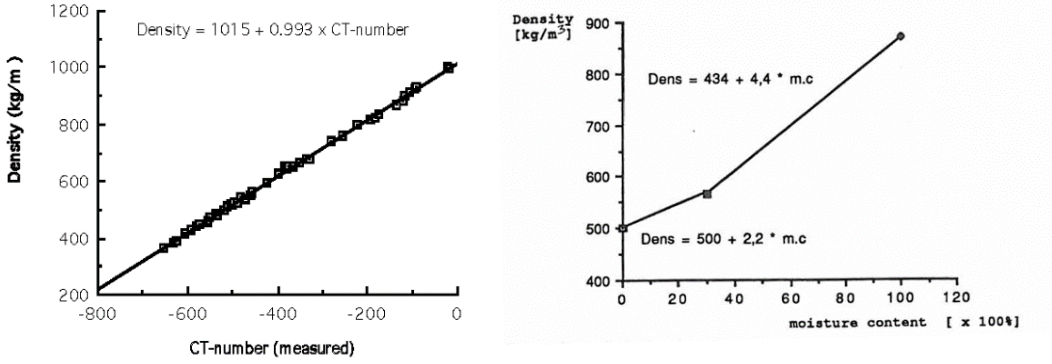

Fig. 3.
Relationship between green wood density and the CT-number /attenuation coefficient (left), and the relation between wood density and moisture content for a sample of wood with a dry density of 500 kg/m^3.

Wood moisture is an important property to be measured and is highly related to the wood density that is a property that can be monitored using CT. One of the potential industrial applications of CT images is the monitoring of wood moisture during the drying process. Fig. 3 shows how the wood density increases with moisture content for a sample of wood with a dry density of 500 kg/m3. Wood swells from 0 to ca. 30% moisture content until the fibre saturation point (FSP) is reached. Above the FSP, the wood cell wall cannot absorb more water and wood stops swelling.

Since the correlation between density and attenuation coefficient is very high (Fig. 3), this relationship can be applied to evaluate "before and after" images using image processing, e.g. with the help of the software ImageJ developed at the National Institute of Health (ImageJ 2010). The data recorded by the CT scanner during the process is converted into a two- or three-dimensional image that, for instance, can show dynamic moisture behaviour during wood drying and crack formation.

THE VIRTUAL STEM BANK

The CT-scanner at LTU has been used to build up a stem bank of scanned trees that can be used for further studies such as anatomical studies, modelling of the sawmill process or the peeling of veneer, wood component processes, and for developing automatic applications for the sorting of logs and sawn timber. The stem bank contains information about 200 Scots pine trees from 33 permanent and well-documented sample plots at the Swedish University of Agricultural Sciences, 144 Norway spruce trees from 44 sample plots in Sweden, Finland and France, and a dozen oak logs. Some of the sample plots have been observed for up to 100 years and the databases include forestry data such as stand data, silvicultural treatments, images from the stand, images of the sampled trees etc.

To be able to use CT-scanned data in different simulations, the amount of data has to be reduced and pixels in the CT images need to be classified according to different types of wood such as knots, heartwood etc. A parametric version of the CT-data has therefore been developed. The outer shape of the logs and the sapwood/heartwood border are described by a radius every degree for a circumference at every 10 mm cross-section in the length of the log, and every knot is described by ten parameters describing volume extension, type of knot, and location in the stem. More details of the stem bank are given in Grönlund et al. (1995) and Nordmark (2005). An example of a log model with outer shape and knots is shown in Fig. 4. Some examples of how the stem bank has been used are:

- The development of stem models that describe how knot properties vary in the stem and how different parameters influence the knot properties.
- An analysis of relationship between log grade and the grade of sawn timber.
- The development of industrial X-ray log scanners, including simulations of X-ray signals and development of control algorithms.
- An analysis of measurement accuracy for different log diameter measurement principles.
- The development of a method for the measurement of spiral grain in CT-images.
- An analysis of different sawing strategies for sawmills.
- The measurement of bark thickness.
- An analysis of resin pocket frequency.
- The development of a heartwood distribution model.

Fig. 4.
Example of log model based on the virtual stem bank

SIMULATION OF THE SAWING PROCESS

In order to study the potential for recognizing the inner properties of logs, a specific simulation software has been developed for the sawing process, Saw2003 (Nordmark 2005). The input is log models, based on the CT scanned logs of e.g. the stem bank. Saw2003 models a sawmill that employs cant sawing with two sawing machines, with curve sawing in the second saw. It is also possible to control positioning of the logs during sawing, green dimensions, machinery etc. Boards are quality graded according to a user-specified grading system, based in principle on the Nordic Timber Grading Rules (Anon. 1997).The sawing simulation leads to virtual boards with information about knots, dimensions, quality, value etc. Saw2003 has been used extensively in earlier research (Nordmark 2005, Moberg and Nordmark 2006, Lundahl and Grönlund 2010, Berglund et al. 2013, Fredriksson 2014).

SIMULATION OF THE REFINING PROCESS

With modern scanning and optimization systems, there is a need to understand the interaction between the biological material and the process of turning it into a product. Otherwise there is a risk that the process will be inefficient with a large amount of waste along the way, especially at the end of a longer production chain when several machine grading decisions are involved before a consumer

product is achieved. This understanding is difficult to achieve with high production speeds and automated grading, since there is little human interaction with the wood material. However, research tools capable of modelling the end result of a production process based on the raw material and quality rules would augment this understanding and help to achieve an integration of the forestry-wood chain.

A simulation tool developed and validated by Fredriksson et al. (2015) is one important step on the way to an integrated approach, since it can be used to link tree or log properties to cross-cut products. This is achieved by using virtual boards from sawing simulation as input. The tool is capable of crosscutting the boards, given a product specification with limits on the size of knots and wane for different board sides. The outcome is value-optimized and based on a value of each product set by the user. It is possible to define both fixed length and flexible length (typically finger-jointed) products, which can be considered simultaneously in the optimization.

Board features are classified as either accepted or rejected depending on a maximum length and width. If quality limits for both length and width are exceeded, the feature is considered as a defect and is cut away. The rest of the defect-free wood is considered accepted and is subsequently used to value-optimize the cutting of products.

Combined with a discrete event simulation tool, of which several are available commercially, the cross-cutting simulation tool can be used to predict how different decisions affect production costs in the entire forestry-wood chain. This can augment discussions between actors in the chain, since the cost involved in any decision can be quantitatively predicted beforehand. Other activities such as splitting, wood moulding and planing remain to be modelled in the same way as cross-cutting. If these were added, a wide range of end products could be considered at an early decision stage, making it possible to decide what to do with each individual log before sawing it. To this can also be added other end uses of wood, such as pulp and paper or energy conversion.

INDUSTRIAL USE OF THE CT-TECHNOLOGY

A sawmill's goal is to produce as much valuable wood as possible from roundwood. Due to natural variability, each log is different, although most of the distinguishing characteristics are visible only after sawing. As a result, the decision as to how best to saw the log is driven mainly by the log´s external appearance, which significantly reduces the ability to exploit the roundwood's true value. For this reason, knowing the real internal characteristics of a log before deciding its use has been only a dream for the sawmills. Tools that can use data from industrial scanning of real logs, where internal as well as external log features are represented in three dimensions, e.g. gamma-ray, X-ray, and CT scanners, have been developed since the 1970s, the first gamma-ray scanning equipment being installed in a sawmill in Sweden.

An X-ray scanning equipment for detecting the inner properties of logs was developed in Sweden in the 1990s, the X-ray LogScanner (Grundberg and Grönlund 1995). This technology was developed from the experience of developing the *virtual stem bank* and the simulation software described above. Nowadays, most advanced sawmills use X-ray scanning to determine the inner properties of logs. The X-ray scanners are typically based on discrete X-ray scanning in 1-4 directions while the log is fed through the scanner (Pietikäinen 1996; Grundberg and Grönlund 1997).

With the help of discrete X-ray scanning in an industrial environment, it is possible to measure properties such as: diameter under bark, amount of heartwood/sapwood, density, knot whorl parameters (volume and distance), average annual ring width, log type (butt, middle, top) and species (e.g. spruce, pine). With CT-scanning it is also possible to measure sawing position, quality of boards, strength and stiffness in-line with the production.

In 2008, the Microtec company developed the first prototype of a CT-scanner for log imaging, using a 180 kV, 10 mA X-ray source, rotating at 2.8 rev/s, and implementing spiral tomography with a small cone angle of 0.5 degrees. An approximate algorithm by Feldkamp et al. (1984), the FDK algorithm, was implemented for the tomographic inversion, but the maximum scan speed was only 5 m/min, much less than desired.

In most modern industrial and medical CT scanners, the X-ray is emitted from the source in a cone-beam geometry, instead of the fan-beam geometry that was previously used. The challenge is the reconstruction of a three-dimensional image from this cone-beam geometry because the object is typically moved through the beam, and either the object or the combination of the detector and the X-ray tube is turning (spiral CT). Several solutions have been proposed for the reconstruction of cone-beam geometries using different approaches (Feldkamp et al. 1984; Kachelrieß et al. 2000; Stierstorfer et al. 2002). Some of them are implemented in commercial medical spiral CT scanners, but all of these reconstruction algorithms are approximate in their nature (Kalender 2006). Although

they work reliably for small cone angles, wider cone angles result in cone beam artefacts, blurring the image (Zhu et al. 2004).

The breakthrough by Katsevich (2001, 2002) was to find and implement an exact analytical reconstruction algorithm, widely known today as Katsevich's Algorithm. The algorithm was subsequently further refined (2004) and solved not only the cone-beam artefact problem, but was also clearly better suited for use in situations where a fast movement or a high pitch in spiral CT scanners is necessary (Zhu et al. 2004), which was an important prerequisite for a high-speed CT log scanner. Prof. Katsevich therefore laid the theoretical foundation for a fast industrial spiral CT scanning method and paved the way for the application of CT scanners operating at full speed in sawmills. This was realized by Microtec in 2012 when the first functional industrial CT log scanner was developed, together with researchers at LTU, the Forest Research Institute of Baden-Württemberg and the SP Technical Research Institute of Sweden. The CT log scanner from Microtec yields a continuous, qualitative and full 3D log reconstruction. The CT data is used to control and optimize the board output by cutting the logs in a way that increases the value of the sawn products by approximately 10% in practice. Under ideal conditions, a theoretical value increase of more than 20% is possible (Fredriksson 2014).

For the industrial CT-scanning technology a number of challenges regarding mechanical constraints, data transfer, safety regulations, image processing, and optimization algorithms have been addressed. Easily installable in any sawmill, the CT Log helps to get the most out of each sawn tree. Currently (spring 2017) five sawmills in Europe and the Americas have a CT Log equipment able to scan and optimize the processing of the logs at each mill.

The first Microtec CT Log equipment in Scandinavia and the first ever installed directly in the saw line for the direct control of the breakdown process will be installed at the Sävar sawmill in North Sweden in November 2017. It is an investment of approximately € 3 million and is associated with the rebuilding of the saw line. The precise scanning combined with the latest band-saw technology from Söderhamn Eriksson/USNR - focusing on thin saw kerfs and high speed - ensures maximum volume and value yield.

CONCLUSION

Many studies have demonstrated that scanning the internal characteristics of each log before breakdown in order to optimize the process would result in a significant increase in the sawmill yield.

The advances of X-ray source technologies and the exact cone beam algorithm developed in recent years have led to an industrial CT scanner for the analysis of each log in a modern sawmill. This new technology and its successful implementation mark the start of a new era in non-destructive roundwood scanning. Ground-breaking research and innovative implementation have opened new horizons, facilitating further optimization of the sawing process for the timber industry. Compared to the product value derived from existing technologies in modern European sawmills, where 3D scanning of the shape of the log is used, a 10-15% increase in value of the output can be achieved. If all the advantages of having access to the internal features of the logs were realized, a theoretical increase of the order of 20-25% is possible. For an average-sized sawmill, an investment in this kind of equipment should have a payback time of not much more than a year.

For example, high-speed scanners will also be pivotal in the further integration of related scientific research into the sawing optimization process and will most certainly stimulate further research in the wood processing area.

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FACTORS AFFECTING VOLUME YIELD IN A FORESTRY-WOOD VALUE CHAIN – A SIMULATION STUDY BASED ON CT SCANNING

Magnus FREDRIKSSON
Luleå University of Technology, Wood Science and Engineering
Forskargatan 1, 93187 Skellefteå, Sweden
Tel: +46 910 585708, E-mail: magnus.1.fredriksson@ltu.se

Olof BROMAN
Luleå University of Technology, Wood Science and Engineering
Forskargatan 1, 93187 Skellefteå, Sweden
E-mail: olof.broman@ltu.se

Abstract

The paper presents the results of a simulation study, where log models based on CT scanned logs of Scots pine (Pinus sylvestris L.*) was used as input material to a computer simulation model of a generic value chain involving sawing, drying, crosscutting and finger jointing. The aim was to investigate which factors that affect the volume yield in the value chain, be it forestal, log-, process- or quality-related factors. The results show that factors related to growth conditions and log size have a large impact on the volume yield in the studied value chain, together with quality requirements on knots. Factors such as sawing positioning and log quality had a much smaller impact. It can be concluded that it is possible to model a forestry-wood value chain, while assessing which input variables affect the result in terms of volume yield, using CT scanning of logs and subsequent computer simulation of the production processes.*

Key words: *computer simulation; CT scanning; material efficiency; multivariate statistics.*

INTRODUCTION

The cost of raw material in a forestry-wood value chain takes up a large part of the total costs (Lindholm 2006). Each process step in the value chain affects the material utilization efficiency and therefore the cost efficiency. Wood has high diversity in its inherent features and the different processes must be able to handle this. Every piece of wood is unique and it is challenging to handle the high variability in the input material. In modern production processes, many of the decisions are being made automatically with the aid of computer systems and data obtained from scanning equipment.

Optimization in a production system is usually done at one production unit at a time, optimizing only that particular unit's performance. This leads to sub-optimization since the whole chain from log to an end product is not considered (Perstorper et al. 1995, Usenius et al. 2007). A system based thinking, where the whole chain is considered when optimizing, is more efficient (Beenhakker 1964, Pulkki 2001).

In a long and complex value chain, it is therefore of interest to identify which factors that affect the total material efficiency. Some attempts have been made, based on live tests where the material has been followed through a physical value chain (Broman and Fredriksson 2012). However, there are downsides of doing tests in an industrial environment, i.e. experimenting with the system itself rather than a model. It is usually expensive, time consuming, and, in the case of wood, does not allow for control of variables on a common data set. The reason for this is that wood has inherent properties that are unique for each tree and log, and that many of the production processes involved are irreversible, so the material cannot be restored to its original state. Even though experimenting with the actual system means that there is no need for validation, modelling is advantageous since tests can be done on the same material but with different process parameters.

In some cases, simulation have been used to assess the effect of forest and log features on a product, however these have mainly studied one process at a time, mainly sawing (Pinto et al. 2005, Lundahl and Grönlund 2010, Stängle et al. 2014). Again, this carries with it a risk of sub-optimization and therefore reduced material efficiency, if the value chain contains several sub-processes.

To avoid sub-optimization it is important to study an entire value chain with all included sub-processes, if possible. In addition, simulation models are useful since they reduce the need for expensive and practically unrepeatable test sawing. This study will demonstrate how simulation

models can be used to study a value chain involving several sub-processes, thus applying a holistic perspective of the value chain.

When identifying factors that affect yield in a value chain, some sort of statistical model is needed. Partial least squares (PLS) regression is a good prediction method when the predictor variables are correlated to each other and when there can be noise in the data (Wold et al. 2010). This is often the case when wood is studied. For visualising and analysing the structure of observations and variables, principal components analysis (PCA) is useful (Abdi and Williams 2010).

OBJECTIVE

The objective was to investigate which factors affect the volume yield of a production value chain that includes sawing of logs, and subsequent crosscutting and finger jointing of the resulting boards. This was to be done using CT scanned logs, computer simulation tools, and multivariate PCA and PLS regression modelling.

MATERIAL, METHOD, EQUIPMENT

To model a wood value chain with various production processes, a generic product was chosen as study case. The end product was finger-jointed boards of Scots pine (*Pinus sylvestris* L.) with cross-section dimensions of 38×100 mm. The studied value chain is described in Fig. 1.

Fig. 1.
The studied value chain and its inherent sub-processes and material.

Roundwood data

This study was based on the Scots pine logs of the Swedish Pine Stem Bank (Grönlund et al. 1995). The stem bank trees, from well-documented sites at different locations in Sweden, have been documented thoroughly regarding tree properties and silvicultural treatments. They were bucked into logs that were scanned with a medical CT scanner (Siemens SOMATOM AR.T) to record internal properties. Knots are described by a parameterized model, which takes into account curvature and the diameter of the knot in two directions, tangential and longitudinal. Each knot is divided into a living and a dead part. Details on the knot model are given by Grönlund et al. (1995) and Nordmark (2005).

Since the aim was to produce boards with dimensions 38×100 mm using a cant sawing pattern, only logs with a top diameter between 130 and 149 mm were used. This means that out of the 628 logs in the Swedish Pine Stem Bank, 118 were used for this study.

Sawing simulation

Sawing simulation was performed using the simulation software Saw2003, developed by Nordmark (2005). The input was log models, based on the CT scanned logs of the stem bank. The log models were constructed by the parameterized knot models and an outer shape description of the log.

Saw2003 models a sawmill that employs cant sawing with two sawing machines, with curve sawing in the second saw, edging and trimming. The latter two are value-optimized according to timber prices and grading criteria. It is also possible to control positioning of the logs during sawing. An example of a log model used in Saw2003 is shown in Fig. 2, with outer shape and knots.

Fig. 2.
Example of log model used in this study.

The sawing simulation results in virtual boards with information about knots, dimensions, quality, value and so forth. Saw2003 has been used extensively in earlier research (Nordmark 2005, Moberg and Nordmark 2006, Lundahl and Grönlund 2010, Berglund et al. 2013, Fredriksson 2014).

All 118 logs were sawn using a cant-sawing pattern, resulting in two centreboards with dimensions 38×100 mm. Only the centreboards were considered in this study. Log rotation and offset was controlled during sawing, rotation was done at ten degrees counter clockwise from horns down, at horns down and at ten degrees clockwise from horns down. At each rotational position, the log was moved in the lateral direction from fully centred to a 10 and 20 mm displacement, respectively. At each position, sawing was made and the resulting virtual boards were saved for further processing. In this way, each log was sawn 3×3 = 9 times. The levels were chosen to achieve a large enough variable span for a visible change in yield, and were based on Berglund et al. (2013) and Fredriksson (2014).

Crosscutting simulation

A crosscutting simulation tool developed and validated by Fredriksson et al. (2015) was used to model crosscutting of the sawn boards that were the result of the sawing simulation. The tool is capable of crosscutting boards, given a product specification with limits on sizes of knots and wane. The outcome is value-optimized, and based on a value of each product set by the user.

Board features are classified as either accepted or rejected depending on a maximum length and width. In case both quality limits for length and width are exceeded, the feature is considered as a defect and is cut away. The rest of the defect-free wood is considered accepted and is subsequently used to value-optimize cutting of products. In this study, only knots and wane were considered since this is the only log feature that is included in the stem bank.

Each board was crosscut with the aim of producing finger-jointed boards, i.e. a variable-length product. The maximum allowed knot size was set at three different levels: 10, 25 and 50 mm. The same thing was true for the minimum and maximum length of the crosscut pieces, the minimum length was set to 130, 170 and 210 mm respectively, and the maximum length to 450, 550 and 650 mm. In this way, each board was crosscut $3^3 = 27$ times. Maximum size of wane was set at a constant level of 5 mm maximum length and 2.5 mm maximum width. The levels were chosen based on an industrial case (Broman and Fredriksson 2015, Fredriksson et al. 2015).

Tested variables

20 variables were studied, 18 predictor variables, and two result variables, *Yield* and crosscutting yield (*CCYield*). The variables can also be classified as controllable or non-controllable, since some of them relate to the nature of the biological material. For instance, the growth conditions of the trees and the log shape variables are non-controllable, while the maximum allowed knot size in the product is controllable. The controllable predictor variables are summarized in Table 1, while the non-controllable predictor variables are summarized in Table 2. In addition to the variables in Table 2, a qualitative variable *Log type* describing the log position in the stem was added (butt- middle- or top-log). *Yield* was calculated for each log as the total volume of sawn and crosscut pieces divided by the log volume, while *CCYield* was calculated as the total volume of sawn and crosscut pieces divided by the total volume of sawn and dried timber from each log. Both variables therefore describes volume yield.

Table 1

Controllable variables tested in this study and their variation range

Variable	Saw rotation (deg.)	Saw offset (mm)	Min. length (mm)	Max. length (mm)	Max. knot size (mm)
Abbreviation	Rotation	SawCenter	MinL	MaxL	KnotDia
Process level (Fig. 1)	Sawing	Sawing	Crosscutting	Crosscutting	Crosscutting
Range	-10 to 10	0 to 20	130 to 210	450 to 650	10 to 50
Number of tested values	3	3	3	3	3

Table 2

Non-controllable variables tested in this study and their variation range

Variable	Abbrev-iation	Process level (Fig. 1)	Max value	Min value	Average
Lat-itude (deg.)	Lat	Tree	65	56	62
Height above sea level (m)	HSeaL	Tree	400	100	248
Site index[a]	SI	Tree	28	16	22
Tree length (m)	TLength	Tree	28	18	22
Age (years)	-	Tree	153	70	106
Log quality[b]	VMF_Q	Log	9	1	3
Top diameter (mm)	TDia	Log	149.6	128.8	137.7
Butt diameter (mm)	BDia	Log	233.8	155	180.1
Log vol-ume (m^3)	LogVol	Log	0.144	0.0567	0.0878
Log length (cm)	LogLen	Log	553	329	426
Bow height (mm)	BowH	Log	42	3	15
Taper (mm/m)	Taper	Log	20.4	4.16	10.1

[a]Tree height after 100 years of age
[b]According to the Swedish Timber Measurement Association, VMF

The number of observations were 118×9×27 = 28674, since there were 118 logs sawn in nine different positions, and the resulting boards were crosscut in 27 different ways.

Statistical analysis

A PCA model was fit to all observations and all variables except crosscutting yield, after scaling of each variable to unit variance and centring on the mean. The PCA model was used to survey which variables were correlated and to give an overview of the collected data. After this, two PLS models were fitted to the same data, but using *Yield* and *Crosscutting yield* as the respective result variables. The PLS model was not intended for prediction of yield, rather to point out important variables in the value chain. The analysis was made using the score- and loading plots of the PCA and the PLS models, together with an analysis of the prediction power and the influencing variables of the PLS model.

For any PCA or PLS model, the goodness of fit is given by the calculated coefficient of determination (R^2) and the goodness of prediction by the Q^2-value. The Q^2-value is based on cross-validation (Martens and Naes 1989). Cross-validation means that *n* models are created, each excluding 1/*n* of the observations when creating a training set to build the model on. Each model can then be tested on the observations that were excluded when building the model. These excluded observations are called the test set. The value of Q^2 represents the proportion of variance in the test sets that is explained by the model. This means that Q^2 is a measure of the model's ability to predict new observations, which are observations that were not included when building the model.

To ensure as high predictability as possible, and to avoid modelling noise, the model was fit by adding principal components until the Q^2 value stopped increasing.

RESULTS AND DISCUSSION

The R^2 of the PCA model was 0.33, and the Q^2 was 0.19. The model was fit using two principal components. The R^2 and Q^2 values were not very high, but the reason for this was mainly the qualitative variable *Log type*. If this variable was removed, the R^2 rose to 0.48 but the main correlations remained the same. Therefore, we present the model with the log type variable included. The loading scatter plot for the PCA model is presented in Fig. 3.

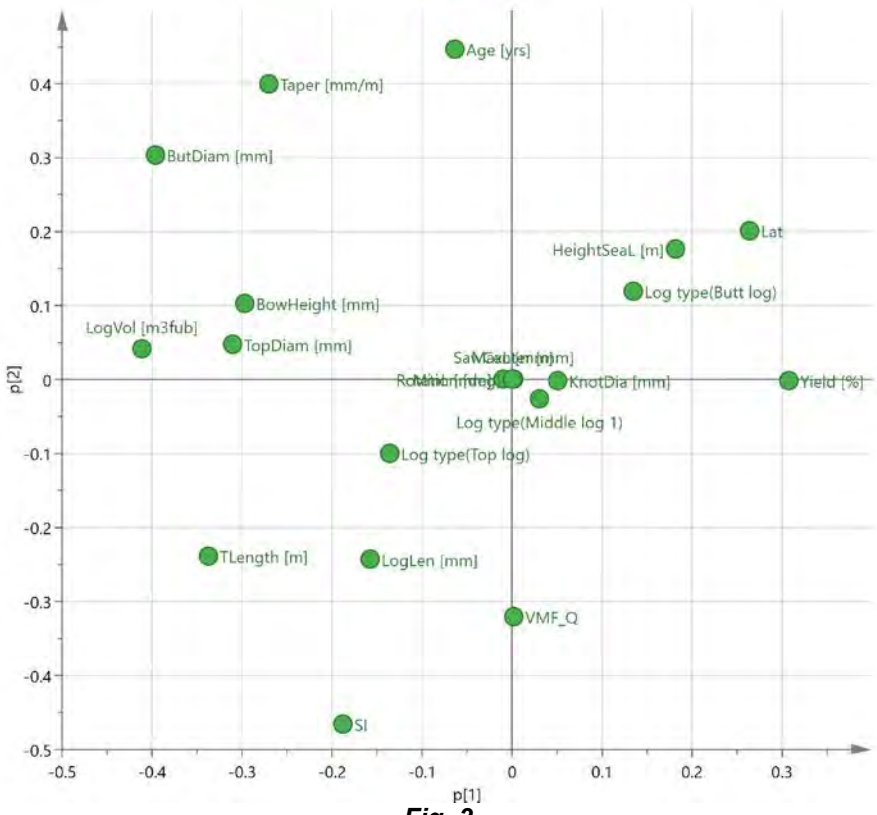

Fig. 3.

***Loading scatter plot of the PCA model, showing principal component 1 (horizontal) and 2 (vertical). The overlapping labels close to the origin are* MinL, MaxL, Rotation *and* SawCenter. For explanations of variable names see Table 1 and Table 2.**

As can be seen in Fig. 3, *Yield* is mainly described by the first principal component, and therefore the other variables' correlation with the yield can be seen as their projected value on p[1]. The strongest positive correlations to yield can be found for variables regarding growth conditions, such as latitude, *Lat*, and height above sea level, *HeightSeaL*, suggesting that a slow growing tree is a more favourable source of raw material in this particular case. This is probably an interaction effect between size of knots, distance between knot whorls, allowed knot size, and minimum length. A designed experiment with this in mind would allow further investigation of these effects.

The strongest negative correlations to yield can be found in variables related to log size and shape, such as top diameter (*TopDiam*), bow height and taper. This suggests that the upper limit of the top diameter range chosen for the sawing pattern might have been a bit too large, sawing logs that were oversized. This also means that taper has a negative impact on yield, since the taper only adds log volume while not radically altering the possibilities for sawing. A large bow height means a reduced yield since curved logs are more difficult to saw than straight ones.

Log type affects the yield indirectly, since butt logs usually have smaller knots, and top logs have larger knots. The log quality as judged by the Swedish Timber Measurement Association had little or no correlation to the yield.

The correlation between the controllable variables and yield is rather small, with the largest effect coming from the crosscutting process. This suggests that the range of sawing parameters were chosen a bit too conservatively, even though they were in line with previous studies.

The score scatter plot for the PCA model is presented in Fig. 4. There are distinct groups in Fig. 4, these are individual logs as can be seen when the score plot is coloured according to the log identifier numbers. Therefore, it is to some extent visible how large the optimization space is for each log, i.e. given a certain raw material, a log, the process itself can be adjusted to increase yield.

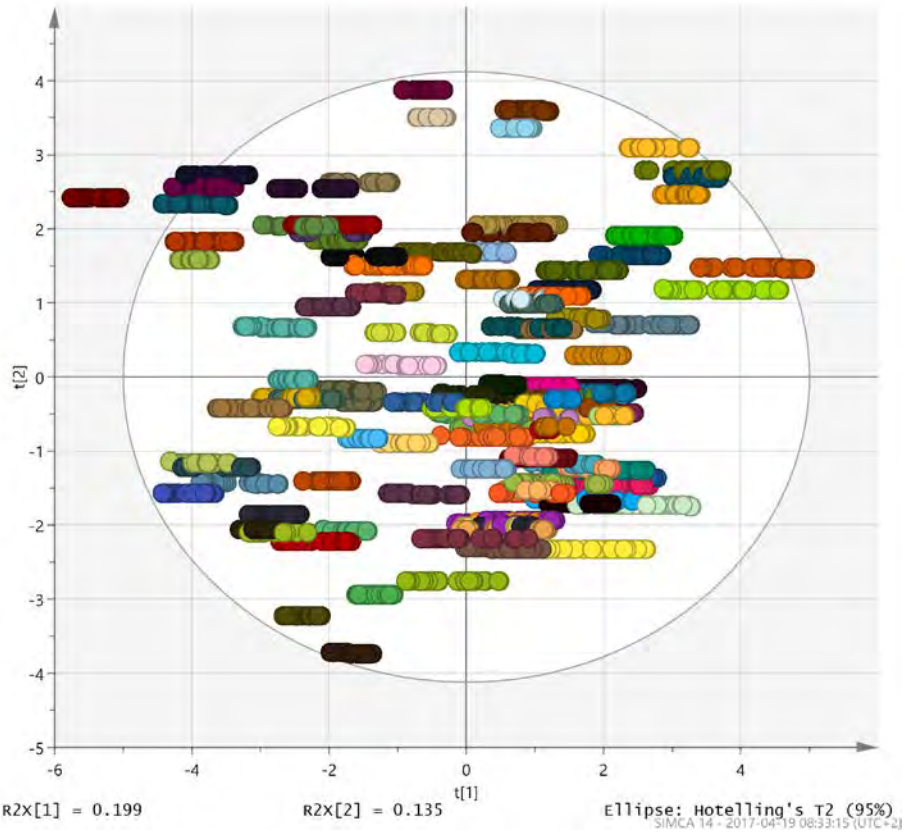

Fig. 4.
Score scatter plot of the PCA model, with the observations colored by log identification number, i.e. each colored group represents one log.

The PLS model using *Yield* as result variable was constructed using three principal components, and had an R^2 and a Q^2 of 0.61. The Root Mean Square Error (RMSE) of the model was 0.067 or 6.7 % yield. Fig. 5 shows a scatter plot with the observed and predicted values of yield for the PLS model. This shows a rather well predicted yield with few outliers, even if there is a fairly large

spread around the unity line. The shape of the plot also suggests that residuals are evenly distributed and centred on zero.

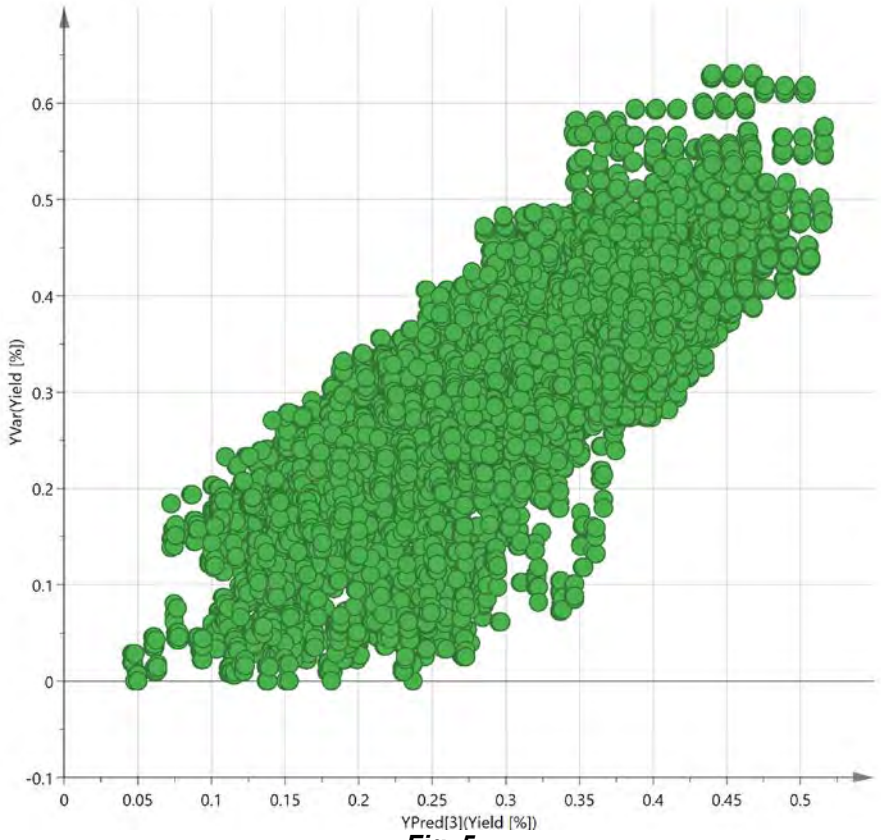

Fig. 5.
Observed (vertical axis) and predicted (horizontal axis) volume yield according to the PLS model.

The coefficients for each of the predictor variables are shown in Fig. 6. The coefficients are scaled and centred to reflect the impact of each variable on the model. The main influencing variable is the maximum allowed knot size. Some of the correlations visible in the PCA model are visible here as well, i.e. variables related to log size, log shape, and tree growth conditions. The minimum allowed length of pieces also has an inverse correlation to yield, since it inhibits the possibilities to optimize crosscutting and fit suitable crosscut pieces between knot whorls.

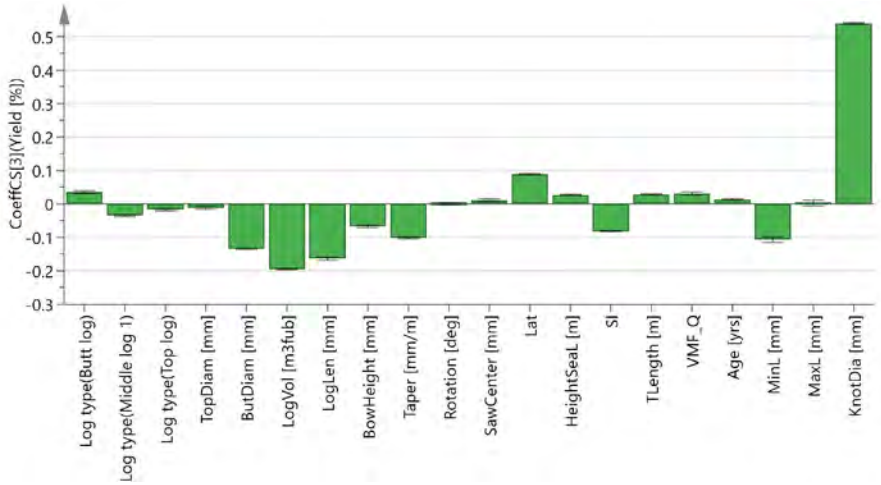

Fig. 6.
Coefficients for the first PLS model, scaled and centred.

The PLS model using *CCYield* as result variable was constructed using two principal components, and had an R^2 and a Q^2 of 0.54. The coefficients for each of the predictor variables are shown in Fig. 7. Many of the effects visible in Fig. 6 can be seen here as well; except that the variables related to the log size and shape have a smaller impact. This is intuitive since *CCYield* only involves the yield in the process of crosscutting boards, so the influencing factors are those related to knots and crosscutting.

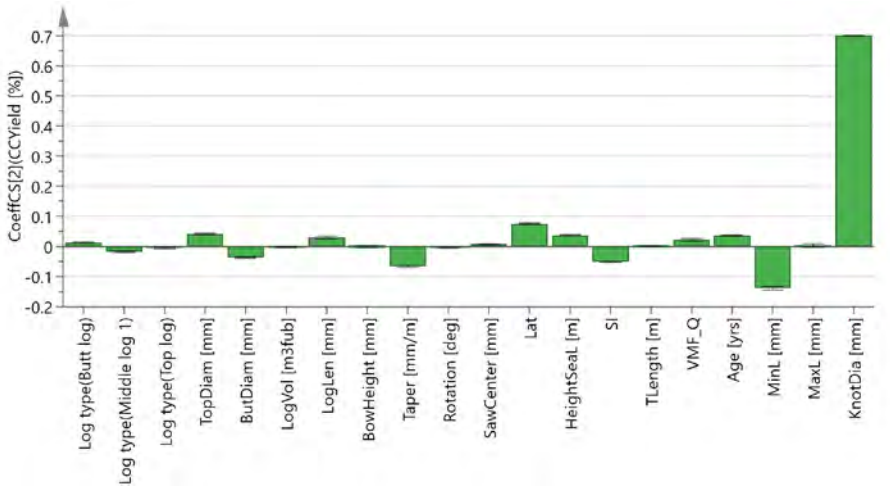

Fig. 7.
Coefficients for the second PLS model, scaled and centred.

CONCLUSIONS

The results show that it is possible to model a forestry-wood value chain, while assessing which input variables affect the result in terms of volume yield. This is enabled by CT scanning of logs together with computer simulation tools and multivariate statistical methods. Future research could be focused on how to use these tools to control processes, finding optimal choices of process variables given a certain raw material for instance.

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PRODUCTION FLOW ANALYSIS BASED ON GT CONCEPTS IN WOOD PRODUTION FACTORY- CASE STUDY

Alketa GREPCKA
Agriculture University of Tirana, Faculty of Forestry Science
Koder Kamez, 1001 Tirana, Albania
Phone: 00 355 67 462 9761, E-mail: agrepcka@ubt.edu.al

Leka GEDESHI
Agriculture University of Tirana, Faculty of Forestry Science
Koder Kamez, 1001 Tirana, Albania
E-mail: lgedeshi@ubt.edu.al

Denisa LAMI (Hajnaj)
Agriculture University of Tirana, Faculty of Forestry Science
Koder Kamez, 1001 Tirana, Albania
E-mail: dhajnaj@ubt.edu.al

Abstract

The wood industry as a processing industry is distinguished for the application of different types of production systems. Globalization force wood enterprises to focus on optimizing technological processes. In our study we have taken a case study in an Albanian company leader in wood industry that is distinguished for the production of over 200 products. The focus of the analysis was PFA analysis based on the concept of GT. System analysis is mainly done in defining the product variety,production sector analysis, production capacity verification, and data collected from technology cards for each machine in the stations. The procedure in production flow analysis started on the population of the parts to be analyzed. In the study we included all the parts in the shop and gathered data from the technological production sheets for each of the part. From the technological production sheets we gathered data on reper part (number of the part used in company as part coding), operation sequence for each of the part, time of operation as well as machine sequence. We consider grouping parts and machines into families and arranging machines in a GT cell using the rank order clustering technique. As result we obtained a rearranged machines into groups which provided more flexibility and optimize the productions of family parts. As conclusion we might say that using the PFA analysis simplify production scheduling, provide more opportunity to each group and obtaining an equilibrium in work flow.

Key words: *flow production analysis; process layout; variety of product; medium quantity production; batch production.*

INTRODUCTION

The wood industry as a processing industry is distinguished for the application of different types of production systems. Among the main factors that should be considered when this industry is taken into consideration are: Types of operations performed; Number of work stations; System layout; Automation and manning; Part or product variety; These five factors determine the directions of development of companies in this industry. Types of operations performed determine processing operations versus assembly operations; Number of workstations define single station versus multi-station system; System layout for more than one station develop variable routing; Manual and semi-automated workstations that require full-time operated attention versus fully automated stations that require only periodic employee attention and part or product varieties define identical work units versus variations in work units that require different processing. We say that wood industry companies are distinguished from the types of activities that they carry out and at high levels (here are included medium and large enterprises). The distinction is made between (1) processing activities on individual labor units and (2) assembly activities of the parts to obtain the final product (Groover 1980). Additional product parameters that play a role in determining the type of production system are: (1) the type of wood material to be processed. Although different sort of wood raw material are used, the machining process is almost the same without making any significant difference. The profound differences will also affect the type of equipment and handling method in the manufacturing system. (2) Part of product complexity which is an important parameter to be considered. In general, the

complexity of the part correlates with the number of processing processes required while the product complexity correlates with the number of components to be assembled to the final product. (3) Part geometry which also determines the type of machinery used for its processing. The distinction is important not only because of differences in the machining processes and machine tools required, but because the material handling system must be engineering differently in two cases rotational and non-rotational parts. In several studies are used different approaches while studying the wood furniture companies. In our study we will consider the Production Flow Analysis (PFA) as an approach to part family identification and machine cell formation that was pioneered by J. Burbidge [Burbidge 1963, Burbidge 1975, Burbidge 1977). It is the method for identifying the part families and associated machine grouping that uses the information contained on production route sheets rather than part drawings. Workparts with identical or similar routings are classified into part families. These families can then be used to form logical machine cells in a group technology layout. Part families are defined by the fact that their members have similar design and\or manufacturing features. There is always a correlation between part design features and the production operations required to generate those features. A production cell designed for the part family would include those machines required to make the composite part (hint: the composite part for a given family is a hypothetical part that includes all of the design and manufacturing attributes of the family. When part families have been determined by visual inspection, part classification and coding or production flow analysis, there is advantage in producing those parts using GT machine cells rather than traditional process-type machine layout. Many quantitative techniques have been developed to deal with problems in a group technology and cellular manufacturing (Askin et al. 1997, Beaulieu et al. 1997, Cantamessa et al. 1987, Chandrasekharan et al. 1987, King 1980, Kusiak 1988). In this study we will consider grouping parts and machines into families and arranging machines in a GT cell using the rank order clustering technique.

OBJECTIVE

The main objective of the present research was to evaluate application of flow analysis based on GT concepts in a wood processing company and defining the advantages of using this method in increasing the competitive advantage of the company in the Albanian and international market.

MATERIAL, METHOD, EQUIPMENT

Site visit to the company was used for data collection. The data collection process lasted for a period of 4 months. System analysis is mainly done in defining the product variety, company classification in the production system frame, production sector analysis, production capacity verification, and data collected from technology cards for each machine in the stations.

Analyses 1: Shop-floor process layout

For research purposes, an albanian furniture production company respectively "Hoxha" Ltd was selected The company is producing furniture in large scale production. The company was established in 1997 in Tirana (the capital of Albania) and in 2008 was transferred to a three-floor building outside Tirana. The company has a wide market share in Albania and with the focus on expanding it abroad. The company is producing bedrooms, chairs, home furniture, doors, kitchen, office furniture etc.The company produce about 200 different products.

The building consists of three floors. On the ground floor are located machines at work stations. The machines are placed based on the process (process layout).

Fig. 1.
Layout of machines in ground floor.

Table 1

List of machines

No.	Machines	Section
1	Press machine	Special purposes
2	Panel saw (edge cutting)	Cutting section
3	Vertical copy milling machine	Cutting section
4	Two side edging machine	Cutting section
5	Band sawing machine	Cutting section
6	Complete planning machine (kombinat)	Ashkelzim section
7	Complete planning machine (kombinat)	Ashkelzim section
8	Drilling machine (CNC)	Drilling section
9	Drilling machine (CNC)	Drilling section
10	Drilling machine	Drilling section
11	Horizontal milling machine	Special purposes section
12	Band sawing machine	Special purposes section

The company is ranked with a medium quantity range and variety of product is soft and for this reason extensive changeover between one product style and the next is not required and therefore is not considered in the production flow analysis of this case.

On the other hand, this soft product variety brings the opportunity to configure the equipment so that groups of similar parts or products can be made on the same equipment without significant lost time for changeovers.

Product complexity is very complicated and we need to undertake qualitative and quantitative aspects. We consider every final product produced in factory and defining indicator of product complexity for each of them . As indicator of product complexity we consider the number of component for each of products. We defined also the part complexity as number of processing steps required to produce it. These data we collected from engineering office and developing each of technological karte and plan of production activities for each part. So we have complexity of assembled products defined as n_p = the number of parts per product and we have processing complexity of each part as the number of operations required to make it as n_o = number of operations or processing steps to make part. Using them allow us to develop relationships between n_p, n_o, P, Q where P refers to total number of different parts or products style. We consider the style of product as P since we are dealing with soft product variety and thus there are only small differences between products.

Table 2

Total number of parts and processing operations

1	Total number of parts manufactured by the company per year (n_{pf})	$n_{pf} = \sum_{j=1}^{P} Q_j n_{pj}$	4,500,000
2	Total number of processing operations performed by the plant (n_{of})	$n_{of} = \sum_{j=1}^{P} Q_j n_{pj} \sum_{k=1}^{n_{pj}} n_{ojk}$	45,000,000

Table 3

Matrix of company due of variety production

$n_o > 1$	**Part producer**	**Vertically integrated company** The company makes all its parts and assembles them into its final products
$n_o = 1$	**Handicraft shop**	**Assembly plant**
	$n_p = 1$	$n_p > 1$

Today's factories are designed with much more specific missions. Referred to as focused factories they are plants that concentrate "*on limited, concise, manageable set of products, technologies, volumes and markets*" (Skinner 1974). It is a recognition that a manufacturing plant cannot do everything. It must limit its mission to a certain scope of products and activities in which it can best compete. As it may seem, from the above matrix the company can aim at its full integration

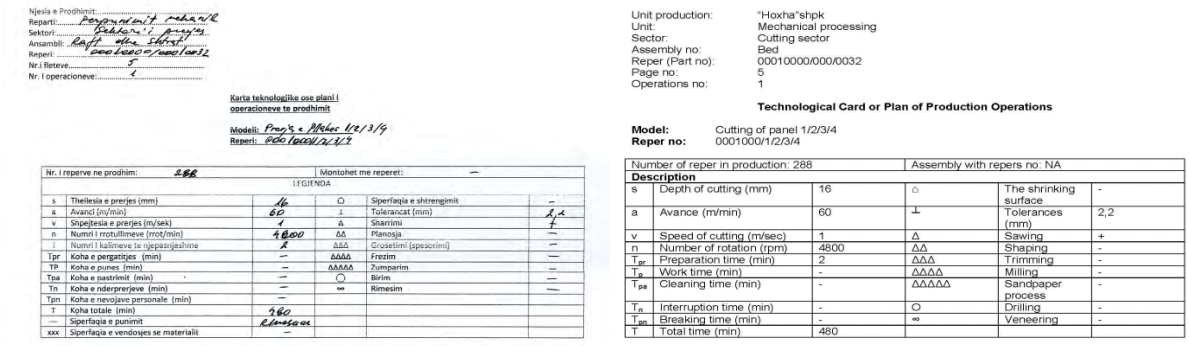
or to pass on its specialization, such as manufacturing only in parts. For this, the management board of the company have to decide what they do not have to do and then focus on it on specifique technologies, products and volumes in which it will finalize. These decisions determine the intendend company's manufacturing capability which three of them are most important: (1) technological processing capability; (2) physical size of product; (3) production capacity.

Analyzing the product variety of the company is a tool prior to the decisions for its specialization. Considering that the technological processing capacity and production capacity are sufficient and the size of the new products does not differ from the previous products we found that the company has the possibility of its full integration for a mass production.

Production flow analysis (PFA)

This method overcome two possible anomalies that can occur in part classification and coding. First, parts whose basic geometries are quite different may nevertheless require similar or even identical process routings. Second, parts whos geometries are quite similar may nevertheless require process routings that are quite different.

The procedure in production flow analysis started based on defined scope of the study which means deciding on the population of the parts to be analyzed. In the study we included all the parts in the shop and collected data from the technological production karte for each of the part. In these cart we gathered data on reper part (number of the part used in company as part coding), operation sequence for each of the part, time of operation as well as machine sequence.



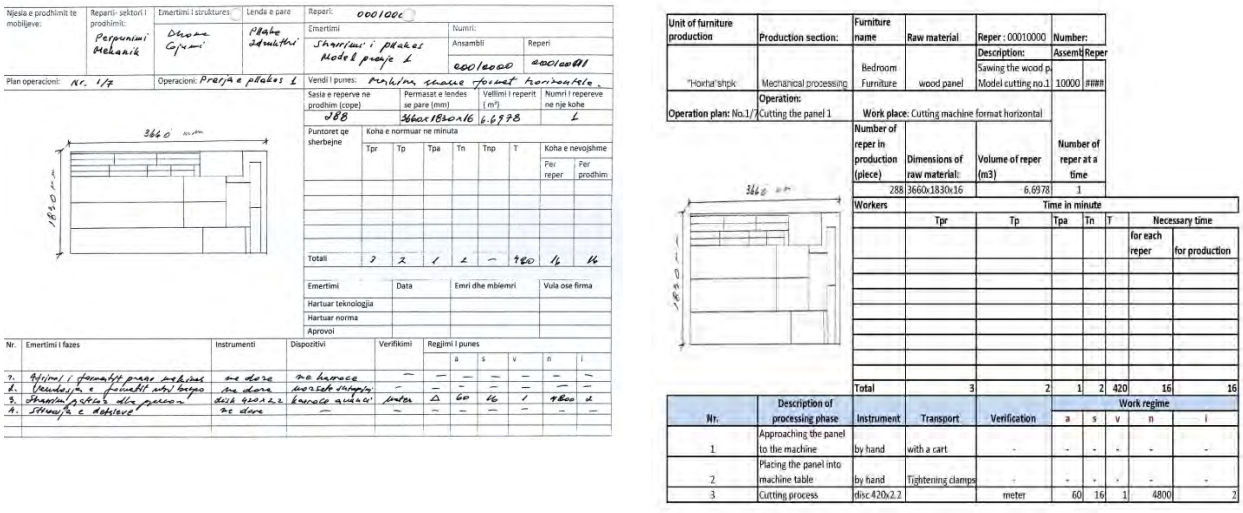
Unit production: "Hoxha"shpk
Unit: Mechanical processing
Sector: Cutting sector
Assembly no: Bed
Reper (Part no): 00010000/000/0032
Page no: 5
Operations no: 1

Technological Card or Plan of Production Operations

Model: Cutting of panel 1/2/3/4
Reper no: 0001000/1/2/3/4

Number of reper in production: 288		Assembly with repers no: NA		
Description				
s	Depth of cutting (mm)	16	The shrinking surface	-
a	Avance (m/min)	60	Tolerances (mm)	2,2
v	Speed of cutting (m/sec)	1	Sawing	+
n	Number of rotation (rpm)	4800	Shaping	-
T_{pr}	Preparation time (min)	2	Trimming	-
T_p	Work time (min)	-	Milling	-
T_{pa}	Cleaning time (min)	-	Sandpaper process	-
T_n	Interruption time (min)	-	Drilling	-
T_{pn}	Breaking time (min)	-	Veneering	-
T	Total time (min)	480		

Fig. 2.
Technological card used for a part.



Unit of furniture production	Production section:	Furniture name	Raw material	Reper : 00010000	Number:
				Description:	Assembl Reper
"Horha"shpk	Mechanical processing	Bedroom Furniture	wood panel	Sawing the wood p. Model cutting no.1	10000 ###

Operation plan: No.1/7 **Operation:** Cutting the panel 1 **Work place:** Cutting machine format horizontal

Number of reper in production (piece)	Dimensions of raw material:	Volume of reper (m3)	Number of reper at a time
288	3660x1830x16	6.6978	1

Workers	Time in minute						
	Tpr	Tp	Tpa	Tn	T	Necessary time	
						for each reper	for production
Total	3	2	1	2	420	16	16

Nr.	Description of processing phase	Instrument	Transport	Verification	Work regime				
					a	s	v	n	i
1	Approaching the panel to the machine	by hand	with a cart	-	-	-	-	-	-
2	Placing the panel into machine table	by hand	Tightening clamps	-	-	-	-	-	-
3	Cutting process	disc 420x2.2		meter	60	16	1	4800	2

Fig. 3.
Technological card used for a part.

Then we follow the second phase of this method which consists in ***sortation of process routings*****. In** this step, we arranged the parts into groups according to similarity of their process routings. To facilitate this step, all operations or machines included in the shop are reduced to code number as shown in table:

Table 3

Code numbers indicating machines and parts for Sortation in PFA (highly simplified)

Parts code	01	02	03	04	05	06	07	08	09	010	011	012	013	014
Machine Code	1	2	3	4	5	6	7	8	9	10	11	12		

On the basis of information gathered by the technological cart of each part, we follow the sortation procedure. The sortation procedure is used to arrange parts in "packs" which are groups of parts with identical routings. Some packs may contain only one part number as part 1 (Table 2) indicating the uniqueness of the processing of that part while other packs will contain many parts and these will constitute a part family. As the result we identified 15 part family and table 3 shown the family part and their respectively technological sequences.

Table 4

Technological sequences of family parts

Family part	Technological sequences
01	$\mathbf{5}$
02	$\mathbf{5 \rightarrow 3}$
03	$\mathbf{5 \rightarrow 3 \rightarrow 8}$
04	$\mathbf{5 \rightarrow 4 \rightarrow 8}$
05	$\mathbf{5 \rightarrow 4 \rightarrow 8}$
06	$\mathbf{5 \rightarrow 4 \rightarrow 9}$
07	$\mathbf{5 \rightarrow 4 \rightarrow 8}$
'8	$\mathbf{5 \rightarrow 4 \rightarrow 10}$
09	$\mathbf{5 \rightarrow 3 \rightarrow 7}$
010	$\mathbf{5 \rightarrow 3 \rightarrow 8}$
011	$\mathbf{5 \rightarrow 3 \rightarrow 4 \rightarrow 8}$
012	$\mathbf{5 \rightarrow 2 \rightarrow 4 \rightarrow 8}$
013	$\mathbf{5 \rightarrow 2 \rightarrow 8}$
014	$\mathbf{11 \rightarrow 6 \rightarrow 1}$
015	$\mathbf{11 \rightarrow 7 \rightarrow 1}$

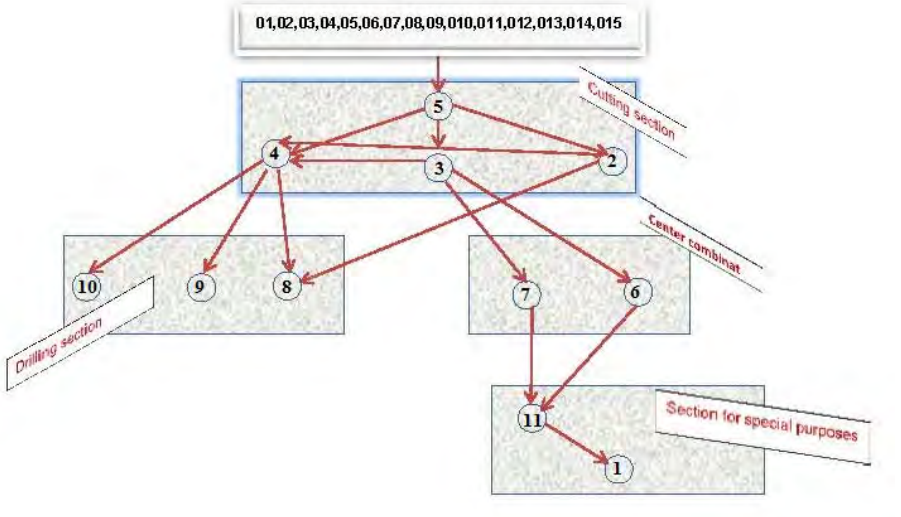


Fig. 4.
Flow diagram of parts in stations.

The processes used for each pack are then displayed in PFA chart. In recent GT literature the PFA chart has been referred to by the term *part-machine incidence* matrix. In the matrix shown below the entries has a value $x_{ij} = 1 \; or \; 0$: a value of $x_{ij} = 1$ indicates that the corresponding part i requires processing on machine j , and $x_{ij} = 0$ indicates that no processing of component i is accomplished on machine j . For clarity in matrix below, the 0's are indicated as blank (empty) entries.

Table 5

PFA chart known as Part – Machine Incidence Matrix. First Iteration (Step 1) in the Rank Order Clustering Technique applied to our case study

	2^{14}	2^{13}	2^{12}	2^{11}	2^{10}	2^{9}	2^{8}	2^{7}	2^{6}	2^{5}	2^{4}	2^{3}	2^{2}	2^{1}	2^{0}		
	PARTS																
MACHINES																	
	01	02	03	04	05	06	07	08	09	010	011	012	013	014	015	Decimal value	Rank
1															1	1	11
2												1	1			12	8
3		1	1						1	1	1					12400	2
4				1	1	1	1	1			1	1	1			3994	4
5	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	32767	1
6														1		2	10
7									1						1	65	7
8			1	1	1		1			1	1	1	1			7484	3
9						1										512	5
10								1								128	6
11														1	1	3	9

Table 6

Second Iteration (Step 2&3) in the Rank Order Clustering Technique applied to our case study

	MACHINES	PARTS															
		01	02	03	04	05	06	07	08	09	010	011	12	013	014	015	Binary Value
	5	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	2^{10}
	3		1	1						1	1	1					2^{9}
	8			1	1	1		1			1	1	1	1			2^{8}
	4				1	1	1	1	1			1	1	1			2^{7}
	9						1										2^{6}
	10								1								2^{5}
	7									1						1	2^{4}
	2												1	1			2^{3}
	11														1	1	2^{2}
	6														1		2^{1}
	1															1	2^{0}
Decimal		1024	1536	1792	1408	1408	1216	1408	1184	1552	1792	1920	1416	1416	1030	1045	
Equivalent rank		15	5	2	8	9	11	10	12	4	3	1	6	7	14	13	

Table 7

Rearranged PFA chart, Indicating Possible Machine Groupings

		PARTS															
		011	03	010	09	02	012	013	04	05	07	06	08	015	014	01
ARRANGED MACHINE IN STATIONS	5	1	1	1	1	1										
	3	1	1	1	1	1										
	8	1	1	1												
	4	1														
	7				1											
	5-2						1	1	1	1	1	1	1			
	9						1	1	1	1	1	1				
	4						1	1	1	1	1	1	1			
	10												1			
	2						1	1						1		
	7													1		
	11													1	1	
	6														1	
	1													1		

GT:1 GT:2 GT:3

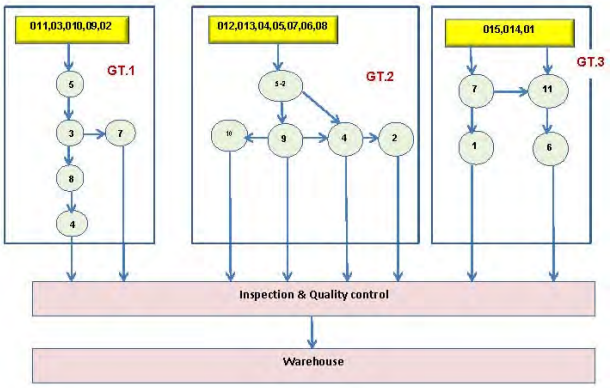


Fig. 5.
Flow diagram of parts in rearranged GT.

Fig. 6.
Rearranged of machines into GT.

The rearrangement of machines into technological groups brought a better flow of parts. Currently we have a clear division of the family of parts of soft varieties into two technological groups. This leads to a better distribution of workload and a better use of manufacturing capacities of all machines. In the above scheme we note a yellow corridor that expresses a flexible workflow path of parts and workers. The third group (GT3) serves for special works and has an interconnection with the other two groups. The two entries in the warehouse provide a direct passage of the final details. This system also provides the possibility of an inspection on every detail machine and final inspection. The weakness of production flow analysis is that data used in technique are derived from existing production route sheets. These route sheets are prepared by engineers and routings may contain some operations which can be unnecessary and consequently the final machining groupings obtained in the analysis may be suboptimal. Eventhough the rearranged flow diagram of parts in GT shows a better route transfer of parts in machines by increasing the opportunity of their quality control.

Conclusion:

1. PFA simplify production scheduling. The similarity found among parts in the family reduced the complexity of production scheduling. Instead of scheduling parts through a sequence of machines in a process – type shop layout, the system simply schedules the parts though cells.

2. PFA provide a clear picture if the availability of machines and provide a distribution of loads amongst machines with same purpose by obtaining an equilibrium in work flow.

3. Grouping machines provides more opportunity to each cell to specialize in producing a smaller number of different parts. This reduces process variability.

4. Grouping the machines reduces setup times. This is accomplished by using group tooling (cutting tools and fixtures etc.) for processing a part of family rather than part tooling which is supposed to be used for an individual part.

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CONTRIBUTION AND COMBINATION OF DIFFERENT WOOD SECTIONS IN SPECIES RECOGNITION USING IMAGE TEXTURE ANALYSIS METHODS

Panagiotis BARMPOUTIS
Faculty of Agriculture, Forestry and Natural Environment, School of Forestry
Aristotle University of Thessaloniki, 54124, Greece
E-mail: panbar@auth.gr

Abstract

The recognition of wood species is a laborious process, which is performed by experts, who attempt to distinguish the different species in different wood sections based on their macroscopic and microscopic characteristics. Most of these characteristics can be observed in the transverse or cross section of woods. According to experts, the next most important surface for wood species recognition is the tangential section while significant information can also be obtained from the radial section of woods. Based on the recent advances in the area of computer vision and pattern recognition, most researchers have proposed image-based approaches attempting to address the problem either in microscopic or macroscopic scale. The main limitation is that in many cases there are some features that are not visible in each wood section. Firstly, we examine the contribution of each section in wood species recognition using two different computer-based texture analysis methods. Furthermore, we compare wood species recognition methods for both grayscale images and colorscale images. Finally, we propose a novel fusion method and we demonstrate that wood species recognition accuracy can be increased by fusing features from different wood sections. For the evaluation of the proposed method, a dataset, namely "WOOD-AUTH", consisting of more than 4272 wood images of twelve common wood species, was used.

Key words: *wood species recognition; wood texture analysis; different wood sections features fusion.*

INTRODUCTION

The recognition of wood species, which is performed by experts, is a laborious procedure because of them having different characteristics, different properties and consequently different money values. In the process of species recognition, the experts classify wood samples by detecting specific characteristics. In any type of operation or activity that uses woods, the species must be carefully considered. In industry, in manufacturing of wood products as well as in construction of residential segments such as roofs, or wood houses the compliance with specific standards is a prerequisite for maintaining security. The correct choice of the desired wood helps a) to ensure the quality of construction and b) to avoid the extra cost of construction.

At each stage of the wooden structures, process recognition is performed by experts and it is primarily based on distinguishing of the macroscopic and microscopic characteristics of woods. First of all, most of these characteristics can be observed in the transverse or cross section of woods. Secondly the tangential section is the next most important surface for wood species recognition and follows the radial section (Bond & Hamner 2002, Jones 2016).

Nowadays, the evolution of imaging technology has positively affected several areas of computer vision. In the case of recognition of species of woods, image techniques based on image processing, texture analysis (Davis et al. 1979) and textural features (Haralick 1979) have significant advantages. Specifically, experts require a long time to train to identify specific wood species while computer based systems training time is short with fast process of recognizing. Furthermore, vision based systems allow simultaneous recognition in different places without extra expenses and with quite good reliability.

Several techniques have been proposed for the recognition of wood species attempting to address the problem either in microscopic or macroscopic scale. In the case of microscopic image analysis researchers aim to identify various microscopic wood features using CCD microscopes and use them as input to the classification system (Cavalin et al. 2013, Yuliastuti et al. 2013). Notable is the method that proposed by Gurau et al. (2013) and based on ImageJ, an image processing program intended for medical microscopy that separate anatomical structures of wood sections and estimate statistical variables. On the other hand, wood recognition approaches based on macroscopic images have recently attracted increased interest, mainly due to their flexibility, simplicity and operability (Hu et al. 2015). Many of wood species recognition use macroscopic grayscale images, co-occurrence matrices (GLCM) and statistical analysis in these images (Tou et al. 2007, Khalid et al. 2008, Wang

2010, Samanta et al. 2015). A similar method for forest species recognition that combines color-based features and GLCM was presented by Filho et al. (2010). Furthermore, two related research works in wood species recognition are presented by Bremanath et al. (2009) that classify ten Indian wood species using the GLCM and Pearson correlation technique. The research approach conducted by Mohan et al. (2014) divides images into blocks using grayscale images. Then, GLCM features are generated from the above blocks and a correlation formula is used for recognition of wood species.

The main limitation, however, of all above approaches is the fact that they take into account features that are derived from a single section of a wood species. In this paper we examine the contribution of each section in wood species recognition and we propose a novel fusion method that combines features of two different wood sections. To this end, we use two computer-based texture analysis methods: a) using co-occurrence matrices and b) introducing linear dynamical systems.

OBJECTIVE

In this paper we examine the contribution of different wood sections in the procedure of wood species recognition using two different computer-based texture analysis methods. Specifically, we examine the wood sections contribution using co-occurrence matrices (CMs) both for the grayscale images and the colorscale (RGB) images concatenating the extracted features of each channel as described by Barmpoutis and Lefakis (2016). Furthermore, this paper, inspired by literature methodology (Barmpoutis 2017), enables the representation of wood images as histograms of LDS descriptors produced by 2-D patches. Finally, we show that wood species recognition accuracy can be increased by the novel combination of two different wood section features.

MATERIAL, METHOD, EQUIPMENT

The proposed methodology consists of several steps, as is shown in Fig. 1. Capturing wood images, it is important to clarify that sometimes, in small wood specimens it is difficult specifically for non experts to distinguish tangential from radial sections. For this reason, we examine the contribution of sections to wood species recognition using a) cross and b) tangential or radial sections. Subsequently, in this paper we propose wood species recognition rates using the combination of two different sections including one cross section and one of tangential or radial section from the same specimen. Furthermore, we compare wood species recognition methods for both grayscale images and colorscale (RGB) images using two different computer-based texture analysis methods: a) co-occurrence matrices (CMs) and b) linear dynamical systems (LDSs).

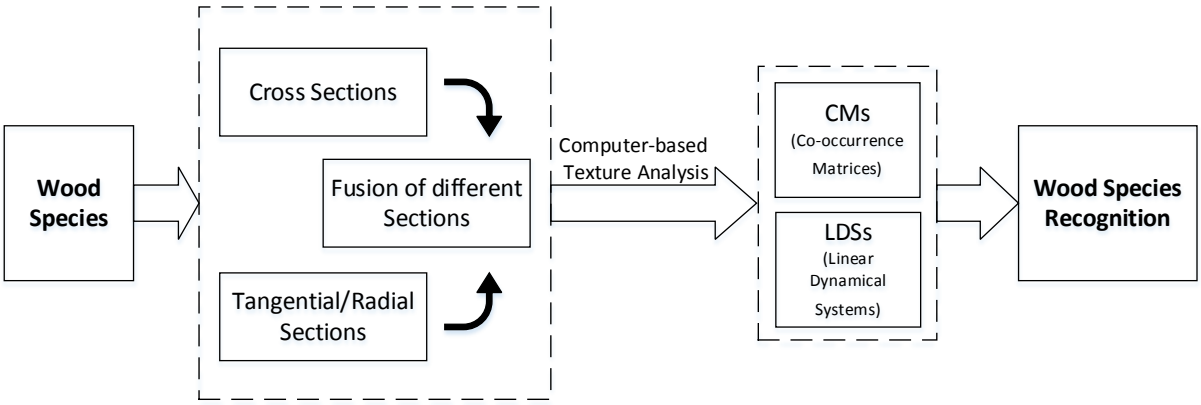

Fig. 1.
Proposed Methodology.

For the purpose of the specific research the database "WOOD-AUTH" was used (Barmpoutis, 2017). This dataset consists of twelve wood species (three softwood species and nine hardwood species) that exist in Greek territory (Tab. 1). The total number of images from cross, radial and tangential wood sections in the dataset is 4272. We have to note that images of this dataset have not been taken by professional photographers and under ideal shooting conditions. Wood images of "WOOD-AUTH" dataset were acquired at the Laboratory of Wood Technology of Forestry and Natural Environment School of Aristotle University of Thessaloniki, Greece and they were taken from distance of 15-20cm using Nikon D3300 digital camera of 24 megapixels. All images size of datset is 400 x 400 pixels.

Table 1

Wood species of 'WOOD-AUTH' dataset

Botanical Name	Category	Botanical Name	Category
Alnus glutinosa	Diffuse-porous hardwood	**Fagus sylvatica**	Diffuse-porous hardwood
Juglans regia	Semi-diffuse-porous hardwood	**Platanus orientalis**	Diffuse-porous hardwood
Ailanthus altissima	Ring-porous hardwood	**Castanea sativa**	Ring-porous hardwood
Fraxinus ornus	Ring-porous hardwood	**Quercus cerris**	Ring-porous hardwood
Robinia pseudoacacia	Ring-porous hardwood	**Cupressus sempervirens**	Softwood
Picea abies	Softwood	**Pinus sylvestris**	Softwood

Texture analysis using co-occurrence matrices and analysis

The use of the co-occurrence matrix is a powerful and widely used texture analysis method. The advantage of the co-occurrence matrix or co-occurrence distribution calculations is that the co-occurring pairs of pixels can be spatially related in various orientations with reference to a) distance and b) angular spatial relationships.

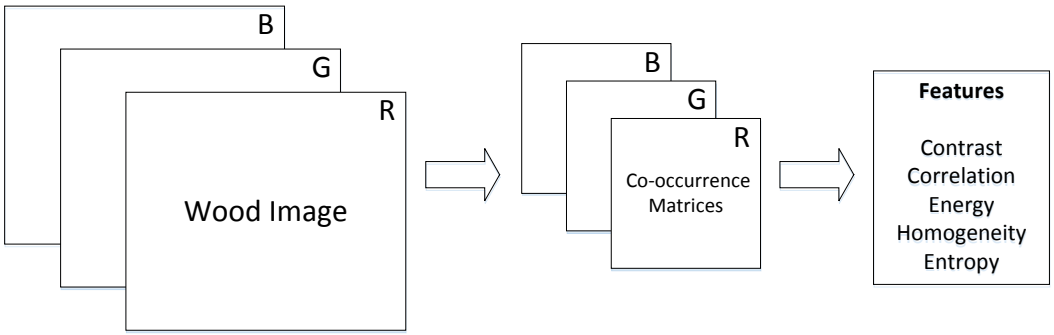

Fig. 2.
Representation of colorscale images (RGB) using co-occurrence matrices and analysis.

In order to use the three channels of RGB images we calculate co-occurrence matrices and features of them for each channel. Co-occurrence matrices are tabulations of how often different combinations of pixel brightness values occur in each channel of an image. To generate co-occurrence matrices we focus on four directions of pixels combinations (0 degrees, 45 degrees, 90 degrees and 135 degrees) and we define the spatial distance of pixels equal to d. For each direction and each channel we extract 5 textural features. The co-occurrence matrices are defined as *G*×*G* matrix for images with *G* values for each channel. Co-occurrence matrices are presented by $C_d(m,n)$ where the first pixel value is m and the second pixel value is *n*. The normalized co-occurrence matrices are represented by:

$$p(m,n) = \frac{1}{\sum pairs \quad of \quad pixels} C_d(m,n) \tag{1}$$

The features that are extracted using each co-occurrence Matrix of colorscale images are the following:

Contrast, for estimation of local variations:

$$Contrast = \frac{1}{(G-1)^2} \sum_{m=0}^{G-1} \sum_{n=0}^{G-1} (m-n)^2 \, p(m,n) \tag{2}$$

Correlation, for estimation of probability of occurrence for a pair of specific pixels:

$$Correlation = \frac{\sum_{m=0}^{G-1} \sum_{n=0}^{G-1} mnp(m,n) - \mu_x \mu_y}{\sigma_x \sigma_y} \tag{3}$$

where μ_x, μ_y, σ_x, σ_y are defined by the:

$$\mu_x = \sum_{m=0}^{G-1} m \sum_{n=0}^{G-1} p(m,n) \tag{4}$$

$$\mu_y = \sum_{n=0}^{G-1} n \sum_{m=0}^{G-1} p(m,n) \quad (5)$$

$$\sigma_x = \sum_{m=0}^{G-1} (m-\mu_x)^2 \sum_{n=0}^{G-1} p(m,n) \quad (6)$$

$$\sigma_y = \sum_{n=0}^{G-1} (m-\mu_y)^2 \sum_{m=0}^{G-1} p(m,n) \quad (7)$$

Energy, for estimation of uniformity:

$$Energy = \sum_{m=0}^{G-1} \sum_{n=0}^{G-1} p(m,n)^2 \quad (8)$$

Entropy, for estimation of the statistical randomness:

$$Entropy = \sum_{m=0}^{G-1} \sum_{n=0}^{G-1} p(m,n) \log p(m,n) \quad (9)$$

Homogeneity, for estimation of the distribution of elements:

$$Homogenity = \sum_{m=0}^{G-1} \sum_{n=0}^{G-1} \frac{p(m,n)}{(1+|m-n|)} \quad (10)$$

Texture analysis using LDSs

A dynamic texture can be considered as a multidimensional signal or sequences of images of moving scenes (Doreto et al. 2003). Recently, a method that uses solutions of linear dynamical systems and extracted LDS descriptors was presented and used by Doretto et al. (2003) Ravichandran et al. (2013), Barmpoutis et al. (2014) and Dimitropoulos et al. (2015). This method has been used in literature for categorizing dynamic textures in video sequences and for fire detection in videos.

By the assumption that a wood image consists of successive, interrelated and repetitive pixels that are particular for each species of wood we take the advantage of the dynamic texture theory and we introduce the descriptor for wood species recognition in static images. In this way the calculated descriptors (LDSs) solving dynamical linear systems contain both appearance and dynamics information. In both cases of grayscale and colorscale wood images, the dynamic texture can be represented with a 2-D matrix. In colorscale images the matrix is implemented if we concatenate the three channels of RGB images as is shown in Fig. 3.

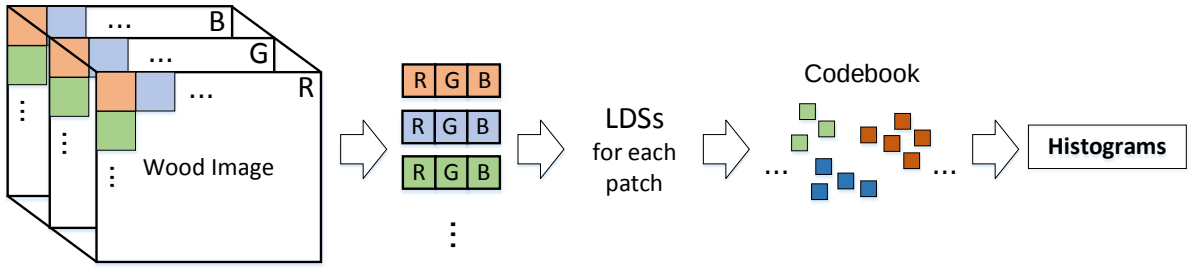

Fig. 3.
Representation of colorscale images using linear dynamical systems.

According to Doretto et al. (2003) the stochastic modeling of both signal's dynamics and appearance is encoded by two stochastic processes, in which dynamics are represented as a time-evolving hidden state process $z(t) \in \Re^N$ and the observed data $y(t) \in \Re^s$, (e.g., *s* indicates the number of pixels in a pacth), as the output of the system:

$$z(t+1) = Az(t) + Bv(t) \quad (11)$$

$$y(t) = C^0 + Bv(t) \quad (12)$$

where: $A \in \Re^{N \times N}$ is the transition matrix of the hidden state, $C \in \Re^{s \times N}$ maps the hidden state to the output of the system, $C^0 \in \Re^s$ is the mean value of pixels intensities, and $w(t) \sim N(0,R)$ and

$Bv(t) \sim N(0,Q)$ are the measurement and process noise, respectively (Doretto et al. 2003, Ravichandran et al. 2013).

Given a wood image *W*, we consider that it consists of non-overlapping patches that contain periodic spatially-evolving (instead of time-evolving data) characteristics whose size is *p×(p×m)*, where *p* indicates the size of patch (in our experiments *p=3*) and *m* is the number of image channels and each patch consists of *s* pixels. If the image is grayscale then *m* is equal 1. To this end, on the assumption that a wood image is considered as a sequence of successive, interrelated and repetitive pixels, it is divided into non-overlapping patches *y*:

$$Y = [y(1) - C^0, ..., y(p) - C^0] = USV^{\mathrm{T}} \tag{13}$$

$$C = U \text{ and } Z = \Sigma V^T \tag{14}$$

The estimated states $Z = [z(1), z(2), ..., z(p)]$ of the system and the matrix A can be computed using least-squares as $A = [z(2), z(3), ..., z(p)][z(1), z(2), ..., z(p-1)]^{pseudoinverse}$, where $Z^{pseudoinverse}$ represents the pseudoinverse of *Z*. Then, each LDS descriptor *M* is defined by *A* and *C*. To solve the problem of wood species recognition there is the need to define the similarity between two LDS descriptors. In the proposed method we adopted the non-Euclidean distance that is based on the subspace angles (Doretto 2003). The calculation of the subspace angles between the two LDSs is performed by first solving for *P* from the Lyapunov equation $A^T PA - P = -C^T C$, where:

$$P = \begin{bmatrix} P_{11} & P_{12} \\ P_{21} & P_{22} \end{bmatrix}, A = \begin{bmatrix} A_1 & 0 \\ 0 & A_2 \end{bmatrix}, C = \begin{bmatrix} C_1 & C_2 \end{bmatrix} \tag{15}$$

The cosine of the subspace angles is calculated by the following formula:

$$\cos^2 \theta_i = i_{th} eigenvalue(P_{11}^{-1} P_{12} P_{22}^{-1} P_{21}) \tag{16}$$

Using the subspace angles, the Martin distance between M_1 and M_2 is defined as:

$$Martin_{distance}(M_1, M_2) = -\ln \prod_i \cos^2 \theta_i \tag{17}$$

The main advantage of LDS descriptors is that they contain both appearance and dynamics information that is represented by *C* and *A* respectively. In the next step we use clustering which is a useful grouping method of data. The LDS descriptors are feeding into K-medoids and using Martin distance we are estimating cluster centers that represent codewords in the codebook. Each wood image in the training set is represented by the distribution of codewords (histograms). Histograms are the features that will be used for classification of each wood specimen.

Concatenation of different section features

Introducing a fusion method that uses both cross and tangential or radial sections we concatanate features that are derived from each of above sections as shown in the following equation:

$$f = [f_T, f_{TR}] = [f_{T1}, f_{T2}, ..., f_{TS}, f_{TR1}, f_{TR2}, ..., f_{TRS}] \tag{18}$$

Because the new feature vector has the double size on comparison to the previous methods, we apply a dimensionality reduction keeping the half components of the feature. The advantages of the above reduction are related to retain to the important components of wood sections features and to hold the computational cost of the proposed method at low levels. The main linear technique for this is the principal component analysis (PCA) that performs a linear mapping of the data to a lower-dimensional space in such a way that the variance of the data in the low-dimensional representation is maximized (Jolliffe, 2002). PCA linearly transform features in order to remove redundant dimensions, and generates a new set of variables called principal components. Using PCA we set the same size to all feature vectors for each method and then they are feed to a SVM classifier.

RESULTS AND DISCUSSION

In this section we present a detailed experimental evaluation of wood species recognition using different wood sections in the "WOOD-AUTH" database (Tab. 2). The goal of the experimental evaluation is three-fold. Firstly, we compare wood species recognition rates using cross sections and

tangential or radial sections. Secondly, we aim to show that the use of two wood sections (cross and tangential or radial) improves wood species recognition. Furthermore, we compare the above implementations using grayscale and colorscale images and two different texture analysis methods. In our experiments we follow the k-fold cross-validation scheme using SVM classifier. The wood species dataset is randomly split into k (in our experiments, k=5) mutually exclusive subsets of approximately equal size. In the k-fold cross-validation, k-1 subsets are used as training data and the remaining single subset is used for testing the model. We repeat the above procedure 5 times.

Table 2

Wood images of 'WOOD-AUTH' dataset.

Contribution and combination of different wood sections using Co-occurrence Matrices

In Fig. 4, we present experimental results using different wood sections and co-occurrence matrices for wood species recognition. As we can notice easily the proposed method using two different wood sections, including one cross section image and one tangential or radial section image, produces the best results. Recognition rates using grayscale images can reach to 74,5% and 83,7% using colorscale images. Experimental results show that there is significant improvement using the original color wood images instead of the grayscale images. This fact really indicates the significance of information that are exist in three channels of colorscale images. It is obvious that when we use tangential or radial sections for wood species classification the recognition rates between the grayscale wood images and colorscale (RGB) wood images are similar, due to the lower importance and quality of characteristics in these sections.

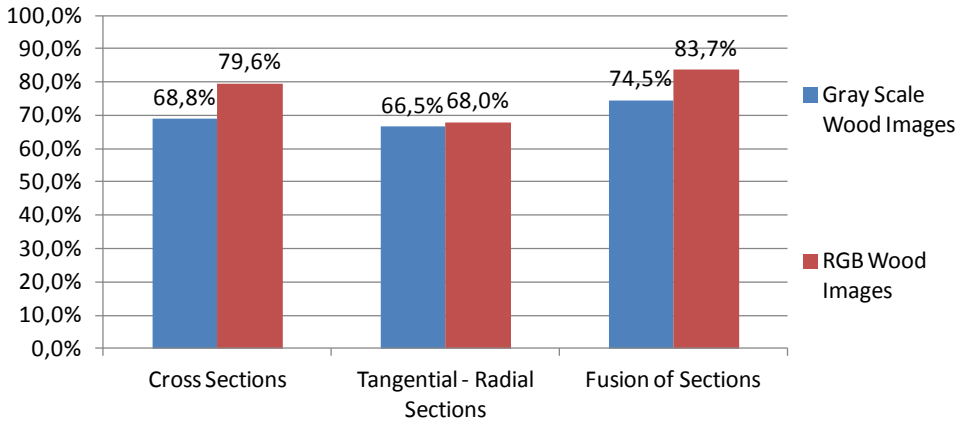

Fig. 4.
Contribution and combination of different wood sections using co-occurrence matrices in grayscale and colorscale wood images.

Contribution and combination of different wood sections using LDSs

By taking advantage of the fact that static wood images contain periodic spatially-evolving characteristics, in Fig. 5 we present wood species recognition results using LDS descriptors, in grayscale or colorscale wood images and in different wood sections. As is it clearly shown, the use of grayscale images of tangential or radial sections produces the lowest recognition rates for both techniques. The classification rate using cross sections and grayscale images is 76,8%, while the recognition rate is lower, 70,6%, using tangential or radial sections. It is worth mentioning that when we use colorscale images and LDS descriptors the recognition rates are slighty increased. They reach to 79% and 72,2% for cross sections and tangential or radial sections, respectively.

It is obvious that the use of LDSs produces in the most cases better results than using co-occurence matrices. The experimental results obtained in this study show the great potential of the proposed method using two different wood sections. Specifically, wood species recognition outperforms with an average true positive rate of 86,1% when we fuse LDS descriptors that are derived both from cross sections and tangential or radial sections. The above fusion using LDS

descriptors shows that it improves significantly the robustness of the algorithm, increasing, however, its recognition ability.

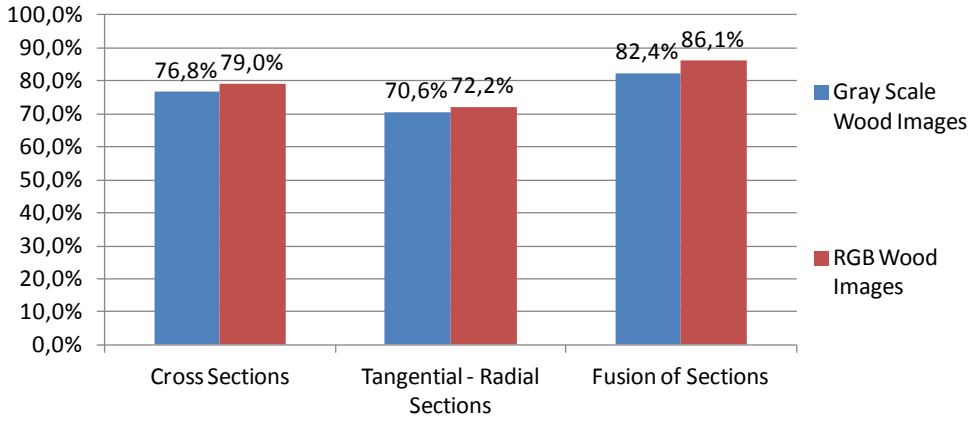

Fig. 5.
Contribution and combination of different wood sections using LDSs in grayscale and colorscale wood images.

CONCLUSIONS

In this paper we presented a detailed comparison and a fusion method for wood species recognition using different wood sections. We used grayscale and colorscale wood images and we compared two different texture analysis methods. In our study we showed that images containing wood cross sections provide higher classification rates than radial and tangential sections. Additionally, the experimental results obtained in this study using the combination of features that are retrieved from two different sections including a cross section and a tangential or a radial section show the great potential of the proposed method. Moreover, in the future we aim to apply the proposed methodology using multidimensional image analysis and higher order systems for texture analysis. Finally, a new challenge for the proposed method would be its application to different wood species.

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INDUSTRY 4.0 – PRAGMATIC ALGORITHMS, INFORMATION QUALITY AND RELATIONAL DATABASES

Timothy YOUNG
University of Tennessee, Center for Renewable Carbon
2506 Jacob Drive
Knoxville, TN 37996-4542
Tel: +01 865 9461119, Fax: +01 865 9461109, E-mail: tmyoung1@utk.edu

Alexander PETUTSCHNIGG and Marius BARBU
Fachhochschule Salzburg GmbH, Salzburg University of Applied Sciences
Markt 136a | 5431 Kuchl | Austria
Email: alexander.petutschnigg@fh-salzburg.ac.at
Email: marius.barbu@fh-salzburg.ac.at

Abstract

'Industry 4.0' is the latest term given to the next significant improvement in manufacturing technologies that is supposed to represent the next great revolution in manufacturing. It has also been called the '*smart factory*,' in which cyber-physical systems monitor the physical processes of the factory and make decentralized decisions. This includes a system of advanced communication networks of PLCs, robotics, and cyber physical visualization and interaction by operations personnel and machines. This will lead to the optimization of manufacturing systems given the changing dynamics of feedstocks, chemical-additives and machines. The goal in this optimization process is a lower variance of process inputs and product quality attributes; that also lead to lower costs of manufactured product. The literature available related to '*Industry 4.0*' addresses the advancements in sensors, robotics, and computational hardware/software systems. However, there is a 'gap' in the literature in the discussion of information quality and advanced database systems that are relational to the sensors and the data from the testing of product attributes. This paper is conceptual and is intended to advance the discussion about the importance of advanced database systems that are relational, and will allow for the development of pragmatic algorithms that are predictive.

Key words: *industry 4.0; pragmatic algorithms; barriers; relational databases.*

INTRODUCTION

We live in a world where electronic transactions for business are common place and occurring at ever increasing rates. Nearly everyone reading this article has a smart phone and most people conduct some type of business with an '*App*' every day – whether it's shopping, searching for information, or summoning a taxi service (Young 2016). Some now define our society as a digital society made of digital citizens. The new generation of people 18 and younger are defined as '*Generation Z*' and are classified as never knowing a world without a smart phone in their hands.

'Industry 4.0' is a name for the current trend of automation and data exchange in manufacturing technologies. It includes cyber-physical systems, the Internet of things, cloud computing and cognitive computing (Kagermann *et al.* 2013, Heiner 2013). '*Industry 4.0*' introduces what has been called the '*smart factory*,' in which cyber-physical systems monitor the physical processes of the factory and make decentralized decisions (Fig.1). The physical systems become '*Internet of Things*,' communicating and cooperating both with each other and with humans in real time via the wireless web (Marr 2016). The idea builds upon the trend in '*Big Data*' and '*Data Mining*,' *i.e.,* the storage and synthesis of large quantities of data for the development of decision support systems. The terms are terms often associated with successful companies such as Google, Amazon, FEDEX, Netflix, etc. '*Industry 4.0*,' was the talk of LIGNA since 2015 and was also among the major themes for the 2015 Woodworking Machinery & Supply Expo in Toronto. Many highly successful companies collect large amounts of data and use statistical algorithms, or "*pragmatic algorithms*" to predict the process outcomes and enhance revenues and improve profitability while focusing on lowering costs (Fig. 2), see Young 2015, Kim *et al.* 2012, Young *et al.* 2014. The financial business sector uses financial engineering to conduct automated business transactions at every increasing speeds.

The forest products industries, while using forward-thinking technologies, has lagged behind the successful implementation of '*Industry 4.0.*' Many in this industry still drive their processes based on the periodic destructive test (or lab sample). Some say "*this is like driving your car looking in the*

rearview mirror." So what are some of the barriers in implementing '*Industry 4.0*' in the forest products industries?

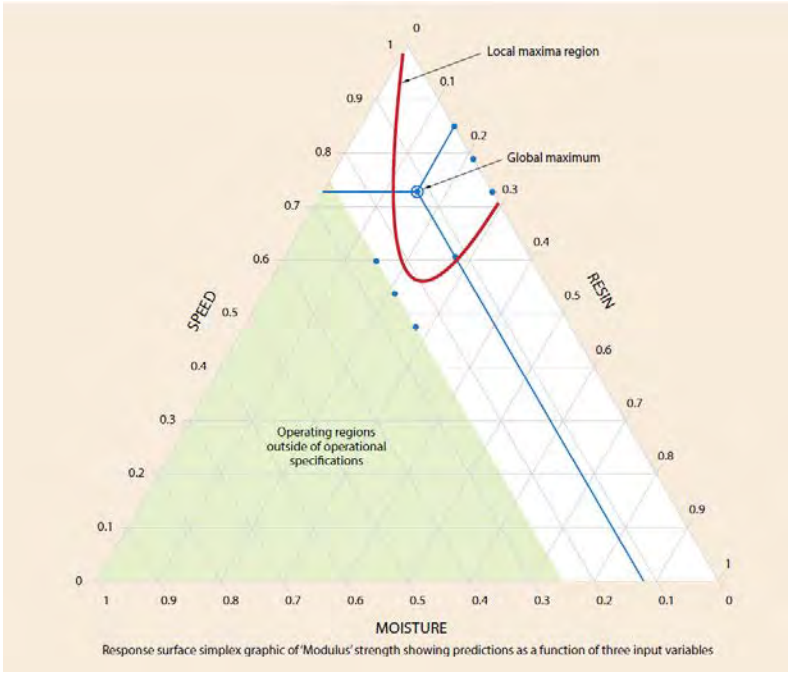

Fig. 1.
Response surface simplex graphic that illustrates optimization.

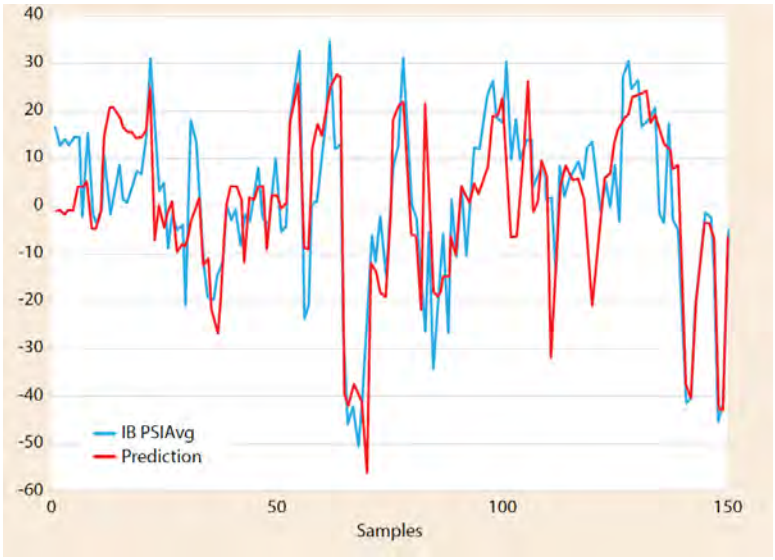

Fig. 2.
Illustration of real-time predictions of "IB" (internal bond) of wood composites.

DISCUSSION

Pragmatic Algorithms and Information Quality

For more than two and one-half decades, forest products plants have used real-time data warehousing to monitor processes. Technical labs have software databases documenting the quality of product. The business functions of accounting and marketing have adopted improved software systems for business transactions, *e.g.,* SAP business software, etc. Control rooms in plants have increased their visualization of data by adding larger and more monitors. Some companies have control rooms that have such a large array of monitors that it resembles a stock exchange or space-agency launch control room (Fig. 3). But in what useful format are the data displayed? Even though we have added more hardware monitors, most are filled with simple trend charts.

Fig. 3.
Illustration of modern operations control room.

Most companies have built smartphone applications for management that allow them to monitor both business transactions and process performance. However, has the increased visualization of data from online sensors led to more effective decision-making by the operations personnel in the control room? Has improved decision-making by operations kept pace with the large databases and advanced analytics? Perhaps it is time for the forest products industries to transition from simple data reporting and rethink how it uses big data and advanced analytics to continually improve competiveness and business performance (Fig. 4), see Carty ***et al.*** 2015, Kim ***et al.*** 2011.

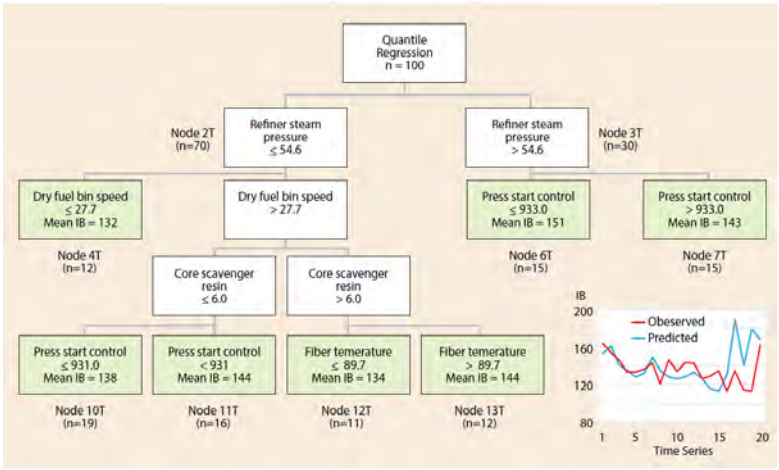

Fig. 4.
Regression tree algorithm with predictions.

A Barrier for 'Industry 4.0' – Absence of a Relational Database

There are several barriers that exist for many forest products companies that inhibit the use of advanced analytics as an improvement tool to improve product quality and lower costs of manufacturing. A key barrier is the creation of a relational database that fuses the data from the process data warehouse with the data from the testing lab. Unfortunately, a key constraint for creating this relational database is that the process data warehouse exists on the process network, and the testing lab database exists on the business network -- separated by a firewall. Even though the software technology for communicating across these networks is trivial, the barrier against this merger of networks is sometimes due to protective corporate polices. Companies like Google, Amazon, FEDEX, Apple, Microsoft, and others, have long ago overcome restrictive company policies that hinder improved decision-making.

Another barrier that must be overcome is improper time alignment of the sensor data that are stored in the process data warehouse (Reigler ***et al.*** 2015). Material flow in the engineered panel manufacturing process from the woodyard to the pressing stage passes across many sensors at different times relative to the time the panel is pressed into a final product. Unfortunately, the process data warehouse sets the time stamp collected from the sensors at periodic intervals (5 minutes) or at delta-change (*i.e.,* only stores data when a value changes). This improper time alignment is a barrier to more advanced analytics and makes it difficult for operations personnel to go beyond basic trend analyses; which are useful, but are subjective and dependent on experience-level. This time alignment problem of sensor data is compounded by the lack of accurate time stamps on panels that are pulled

from the production line for testing. Both of these problems are easily overcome by use of software technology that aligns the process data in time as a function of the line speed or pressing cycle. Accurate time stamping of lab test boards can easily be automated at the sampling location on the process line.

A relational database of the process and lab data can result in an immediate and significant improvement in knowledge of sources of variation by highlighting new data patterns and establishing a platform for predictive capabilities through analytics (Zeng ***et al.*** 2016). Improved knowledge of sources of variation and enhanced predictive capabilities can result in less reject product, a reduction of order rescheduling, lower manufacturing costs, and optimal operations targets for weight, resin, etc.; all of which result in improved business performance.

CONCLUSIONS

Once a relational database has been created, it offers many potential benefits for the organization. Given the advanced capabilities of analytical software and its relatively low cost, improved knowledge of the process can be gained through data visualization and correlations which may also lead to more advanced predictive statistical models of key process parameters (time to closure, weight variation, etc.) and product quality attributes (CSL, modulus, thickness swell, etc.). This goes far beyond trend analyses, and offers additional "root-cause analysis" benefits to enhance statistical process control analyses. Advanced analytics is a business strategy for successful companies of the future. Many companies outside the engineered wood panel industry now have executive positions within the organization to drive analytics-based decision making throughout the organization.

So why not create a relational database of your process? The cost is relatively low given that once the software code is created; the data fusion process is automated. A relational database of key process parameters and product attributes opens up a panacea of benefits for advanced analytical analysis. Advanced analytics is a gateway for lower costs of manufacturing, improved product value and overall improved business competitiveness.

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SECTION 10.

RENEWABLE ENERGY FROM WOODEN BIOMASS

FABRICATION OF ACTIVATED CARBON USING TWO-STEP CO-PYROLYSIS OF USED RUBBER AND LARCH SAWDUST

Jing GENG
College of Material Science and Technology, Beijing Forestry University
Beijing 100083, China
E-mail: eliza2012@bjfu.edu.cn

Jian-Min CHANG
College of Material Science and Technology, Beijing Forestry University
Beijing 100083, China
E-mail: cjianmin168@126.com

Li-Ping CAI
Mechanical and Energy Engineering Department, University of North Texas
TX 72076, USA

Sheldon Q. SHI
Mechanical and Energy Engineering Department, University of North Texas
TX 72076, USA

Abstract

This study was aimed at exploring the characteristics of the char produced in the co-pyrolysis of used rubber and larch sawdust, and the conversion of low-valued pyrolysis char into value-added activated carbon using two-step co-pyrolysis, namely, pyrolysis and activation processes. The physicochemical characteristics of chars were examined by the X-ray diffraction (XRD), Brunauer–Emmett–Teller (BET) and Scanning electron microscopy (SEM). Results revealed that, after the two-step co-pyrolysis, the upgraded carbon had BET surface areas ranging from 600 to 900 m2 g−1, which were higher than the requirements of activated carbon (American Water Works Association B600 standard). It was also found that, as the sawdust/rubber ratio increased, the BET value increased accordingly. The reaction mechanism was proposed based on the experimental results during activation process.

Key words: *used rubber; larch sawdust; co-pyrolysis; activated carbon; BET surface value.*

INTRODUCTION

Used rubber/tires and waste biomass are important fossil-based wastes and bio-based wastes, respectively (Ucar and Karagoz 2014). Approximately 1.5 billion tires are produced and over 800 million tires are scrapped around the world annually (Williams 2013; Beukering and Janssen 2001). The total amounts of above-ground biomass in forests are estimated about 420 billion tons and the yearly global forest residues are increasing gradually (Parikka 2004). Hence, the effective disposal of these wastes has fallen under the category of attractive topics recently. The utilization of waste rubber and biomass through co-pyrolysis is a cost-effective technology to address this issue.

Co-pyrolysis is a promising solution for converting used rubber into value-added products by adding waste sawdust (Laird et al. 2009). Numerous researchers (Zhang et al. 2015; Martinez et al. 2014; Dorado et al. 2014) conducted studies on the improvement of pyrolysis oil obtained from co-pyrolysis of wastes, such as biomass, used tires, waste plastics, coal and sewage sludge. And Shen (2015) reviewed that the low valued char as the main pyrolysis product has the potential to be converted to value-added activated carbon. The American Water Works Association (AWWA) B600 standard requires that the activated carbon has a specific surface area (SSA) in excess of 500 $m^2 g^{-1}$ determined by N_2 adsorption at 77 K (Xia et al. 2016a, b, c, d).

Some researchers have investigated the utilization of pyrolysis char to prepare activated carbon. BET surface areas of poplar wood char from gasification in a fluidized bed were examined by Klinghoffer et al. (2012). It was reported that when the treatments were in gasification atmospheres of 90% H_2O/10% N_2 at 750°C for 60 min and at 920°C for 30 min were used, 435 $m^2 g^{-1}$ and 687 $m^2 g^{-1}$ BET surface areas were achieved, respectively. Carriera et al. (2012) investigated the potential of vacuum pyrolysis to convert sugar cane bagasse into char materials for wastewater treatment and prepared an adsorbent for methylene blue adsorption with a BET surface area of 418 $m^2 g^{-1}$. Bernardo

et al. (2012b) also investigated the co-pyrolysis of plastic wastes, pine biomass and used tires, and achieved activated carbon with an excellent surface area and could be used as precursor for adsorbent.

OBJECTIVE

In our previous work (Wang et al. 2014), the one-step co-pyrolysis of rubber/sawdust was carried out to improve the quality of pyrolysis oil. This work was aimed at exploring the characteristics of the char produced in the one-step co-pyrolysis of rubber/sawdust and the conversion of low valued rubber/sawdust into value-added activated carbon using two-step co-pyrolysis, namely, pyrolysis and activation processes. It is expected that this study is beneficial to reveal the composition of the co-pyrolysis char and the conversion mechanism of the co-pyrolysis char to activated carbon.

MATERIAL, METHOD, EQUIPMENT

The rubber particles with an average size of 80-90 screen meshes were purchased from Ketai Rubber Scrap Mill, Tianjin, China. Sourced from Small Xing-An Mountain, China, the larch wood was dried and smashed into particles. Using a screen, particles with an average size of 80-100 screen mesh were sorted out for the experiment. The composition of the rubber and sawdust was determined (Wang et al. 2014) and is presented in Table 1.

Table 1

Ultimate and proximate analyses of used rubber and Larch sawdust

Material	Ultimate analysis (wt.%)					Proximate analysis (wt.%) [b]			
	C	H	O [a]	N	S	M	VM	FC	A
Rubber	84.10	6.69	7.21	0.39	1.61	4.70	54.53	26.28	14.49
Sawdust	53.40	4.64	41.85	0.11	0.00	10.94	71.59	16.68	0.79

[a] by difference.
[b] air-dried basis.

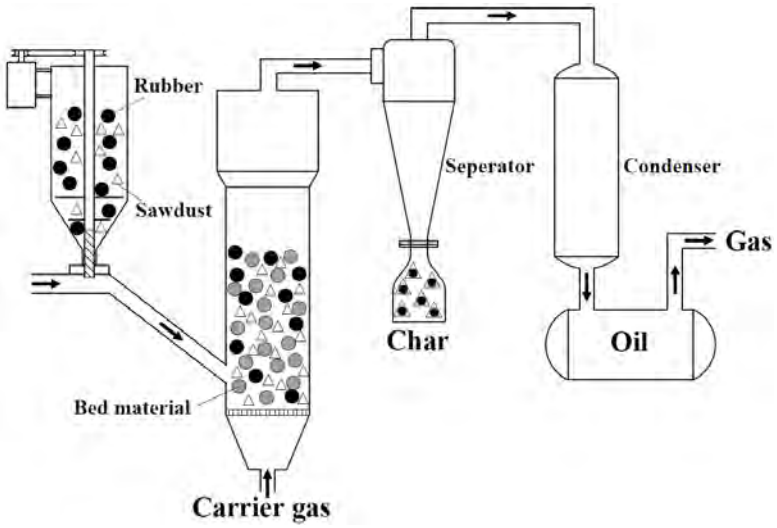

Fig. 1.
The 1 kg h^{-1} stainless pyrolysis reactor.

A laboratory-made 1kg h^{-1} stainless pyrolysis reactor (Fig. 1) with adjustable reaction temperature, heating rate, retaining time and catalyst type was used for the pyrolysis. Under a nitrogen atmosphere, the rubber particles mixed with sawdust were placed in a reactor that was pre-heated at 450°C for 1.2s. The initiation of primary pyrolysis reactions at this temperature released volatiles and formed char. The char was collected by a tourbillion separator, and then the hot volatiles were condensed into pyrolysis oil and non-condensable gas.

The rubber and the sawdust were mixed with different blending ratios (w/w) of 1:2, 1:1 and 2:1 and then pyrolysed in the reactor (signed as 1:2, 1:1 and 2:1). In order to explore the mechanism, the

rubber and the sawdust were pyrolysed separately to obtain the rubber char and the sawdust char as control groups.

The chars obtained in the pyrolysis experiments were carbonized particulate residues impregnated with the pyrolysis oil. Using the Soxhlet method, a solvent extraction (CH_2Cl_2) was performed for 6h. In order to decrease the ash content of the chars, a demineralization procedure was conducted with 1 M HCl for 1h. A KOH activation procedure (the second co-pyrolysis process) was carried out to increase the surface area of the chars. The chars were impregnated with the activator of KOH with the ratio of 1:1, and then activated with N_2 of 100mL min^{-1}. The activation temperature was set as: the temperature increased from room temperature to 500°C with 10°C min^{-1}, and maintained the temperature for 20min; then increased to 800°C with the same heating rate, maintained the temperature for 20min, and reduced to room temperature. The activated chars were washed with 0.1 M HCl and deionized water, and oven-dried to a constant weight. The ash content of chars was detected by the ASTM D 1726-84 Standard "Test Method for Chemical Analysis of Wood Charcoal".

The X–ray diffraction (XRD) patterns of original samples and chars were examined by the Shimadzu XRD–6000 and collected from 5-45° using Cu Kα radiation (60 kV, 80 mA) at a scanning rate of 2º min^{-1}. The specific surface areas of the chars were measured in a Micrometrics TriStar II 3020 (USA) by nitrogen adsorption. Each sample was outgassed for 3 h at 77 K and the surface area was calculated by the Brunauer–Emmett–Teller (BET) method. The surface morphology of the chars was investigated by a Hitachi S-3400N scanning electron microscopy (SEM).

RESULTS AND DISCUSSION

Yield of pyrolysis char

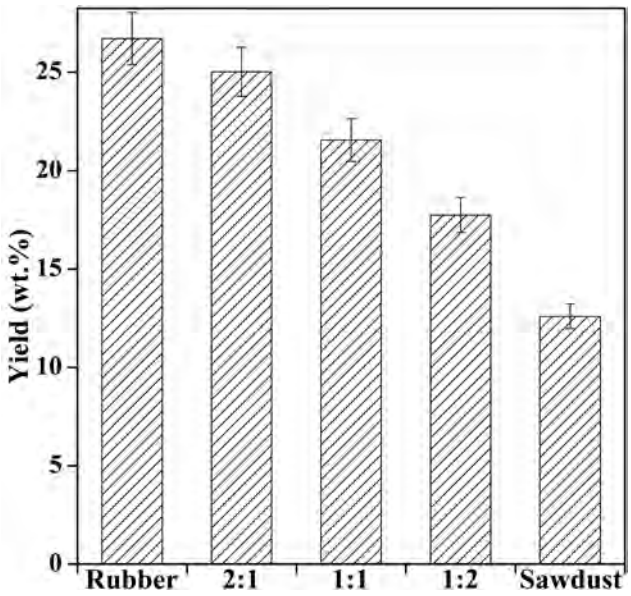

Fig. 2.
Yield of char from co-pyrolysis of rubber and sawdust. 1:2, 1:1 and 2:1 represented the char obtained from co-pyrolysis of rubber and sawdust with different blending ratios (w/w).

As shown in Fig. 2, the char yield of solo rubber pyrolysis was more than twice the amount of the solo sawdust because of the higher content of non-volatile materials like carbon black and ash in rubber [3] as shown in Table 1. As the proportion of sawdust went up, the yield of co-pyrolysis char dropped. In our previous work (Wang et al. 2014), it was also observed that the lower the proportion of rubber, the higher the yield of pyrolysis oil from co-pyrolysis of rubber and sawdust. It was revealed that adding more rubber inhibited the formation of condensable volatiles while promoted the production of solid product during the co-pyrolysis process.

Yield of activated char after upgrading treatments

The preparation of activated carbon with pyrolysis char could increase the added value of pyrolysis char. The cooled process of pyrolysis char in the presence of pyrolysis volatiles would adsorb PAHs and other aromatic molecules (Bernardo et al. 2010; Bernardo et al. 2012a).

Consequently, the decrease of PAHs in co-pyrolysis oil of rubber and sawdust observed in our previous work was not only related to the oxygen free radical reaction during the co-pyrolysis process (Wang et al. 2014), but also affected by the adsorption of co-pyrolysis char.

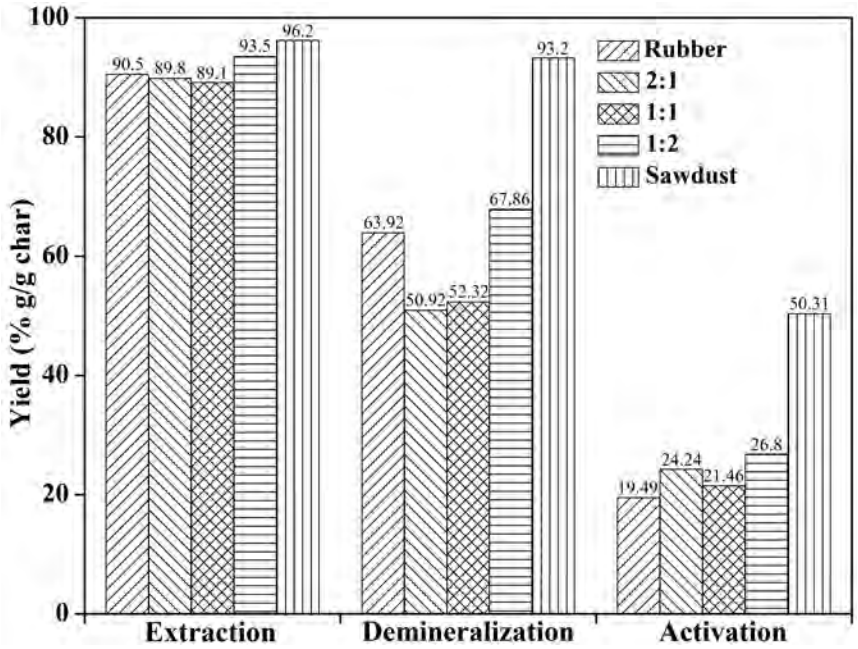

Fig. 3.

Yield of activated char after procedures of extraction, demineralization and activation.

Prior to the KOH activation process, the pyrolysis chars were subjected to extraction and demineralization. Approximately 10% residual oil, which was absorbed by the solid char during the pyrolysis process, was extracted by CH_2Cl_2 observed in Fig. 3. The extraction yield and the demineralization yield of solo pyrolysis rubber char were all higher than those of solo pyrolysis sawdust char. As more sawdust was added in rubber, the char yield after procedures of extraction and demineralization fundamentally decreased and then rose, and the lowest values were 89.1% (1:1) and 50.92% (2:1). It could be inferred that the co-pyrolysis char could adsorb more ash and residue oil, which was very stable aromatic rings in sawdust and long-chain of C–C bonds in the copolymer of rubber and recombination of thermal cracking products during co-pyrolysis process (Laird et al. 2009).

After the activation process (the second co-pyrolysis process), the activated char prepared from solo pyrolysis sawdust char presented high yield after a series of treatments, and the co-pyrolysis char obtained from adding rubber in sawdust was unfavorable to obtain high activated char yield and the activated char yield of rubber char was as low as 19.49%.

Table 2

Ash content of char before and after demineralization and activation

Char sample	Ash (wt.%)		
	Before demineralization	After demineralization	After activation
Rubber	28.70	13.19	16.18
2:1	22.68	7.72	10.36
1:1	23.68	8.03	10.02
1:2	18.16	5.30	7.38
Sawdust	10.02	4.14	5.96

As seen in Fig. 3, the demineralization yield showed that much ash was desorbed. In order to understand the variation of ash in char, the ash content of pyrolysis char was presented in Table 2. It could be seen that the ash content of char depended substantially on the feedstock. The rubber char had high ash content, but the char derived from sawdust showed lower ash content. Through different treatments, ash content of co-pyrolysis char was between rubber char and sawdust char. After the HCl demineralization and KOH activation, the ash content in char decreased significantly compared with that in char without demineralization. The ash content of activated char was slightly higher than that of the demineralized char, which may be related with the activation method and the low activated char yield.

Specific surface area analysis

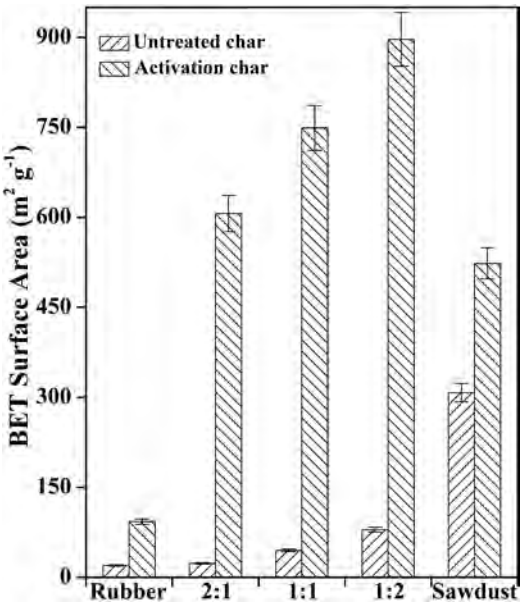

Fig. 4.
BET surface area of char from co-pyrolysis of rubber and sawdust.

The BET surface area of char plays an important role in influencing on its adsorption performance (Guerrero et al. 2008). As shown in Fig. 4, all untreated pyrolysis char showed low values of BET surface areas except for the sawdust char. Even after the KOH activation, the value of surface area in rubber char was too low to be used as activated carbon because the high ash content and carbon black in rubber may block the incipient porosity (Bernardo et al. 2012b; Raveendran and Ganesh 1998; Helleur et al. 2001). When the proportion of sawdust in the mixture increased, the BET surface area of the co-pyrolysis char also increased. After the activation process (the second co-pyrolysis process), the surface area of the co-pyrolysis char reached 896.4 m^2 g^{-1} when the blending ratio of rubber/sawdust was 1:2, which was much higher than the sawdust char (523.1 m^2 g^{-1}) and the rubber char (92.8 m^2 g^{-1}). It could be inferred that the co-pyrolysis char had more extraction yield and demineralization yield so that there would be more pores in char after the extraction and demineralization, which promoted the activation reaction to obtain much cleaner activated char with higher BET values. The obtained BET surface areas were higher than those BET values (from 429 to 687 m^2 g^{-1}) obtained from poplar wood by Klinghoffer et al. (2012).

Mechanism of activated char formation

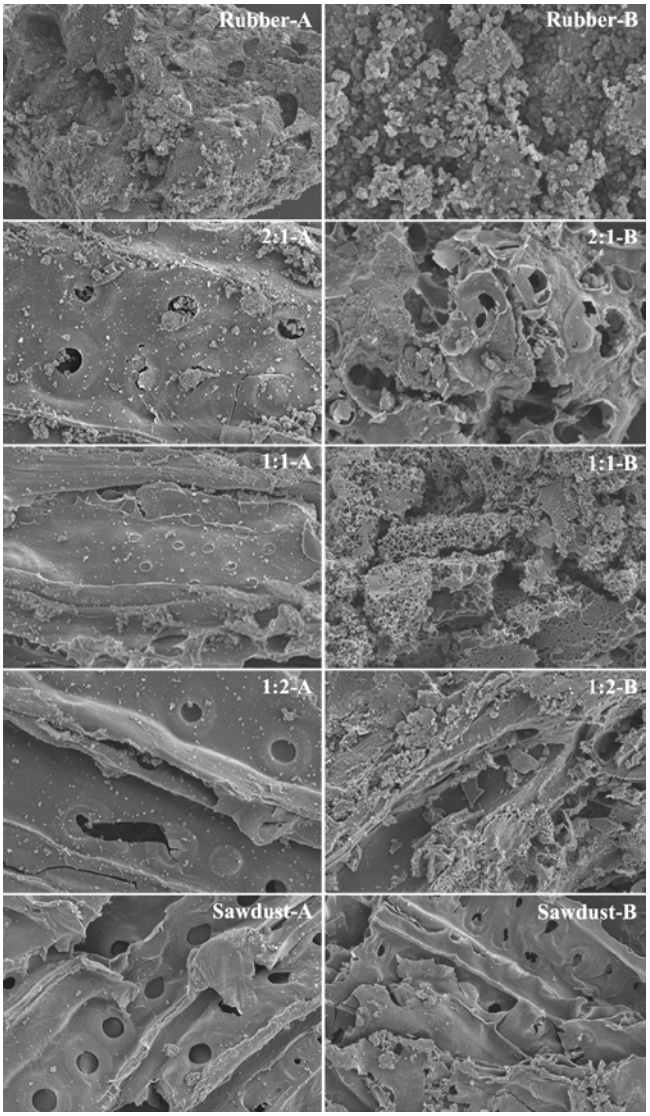

Fig. 5.
SEM images of the char from co-pyrolysis of rubber and sawdust. A: untreated char; B: activated char.

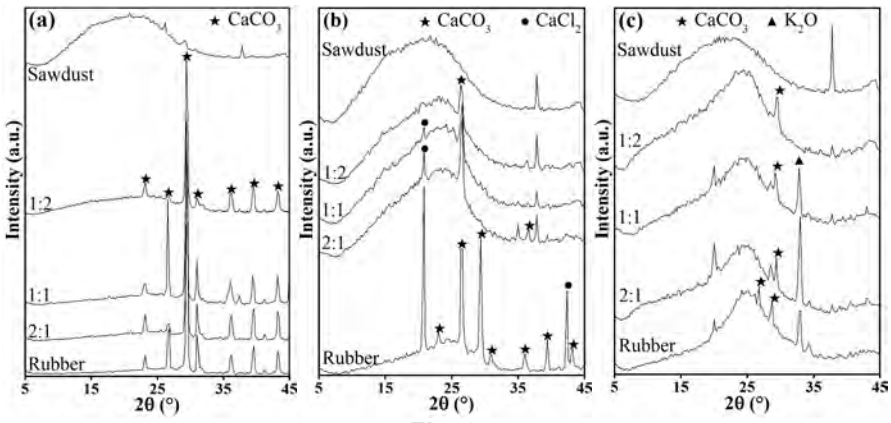

Fig. 6.
X–ray diffraction of the char from co-pyrolysis of rubber and sawdust. (a) Untreated char; (b) Demineralized char; (c) Activated char.

As shown in Fig. 5, some rubber char in the untreated co-pyrolysis char was attached to the surface of sawdust char well and some had entered into the pits of the sawdust char (Fig. 5, 2:1-A). After the activation process (the second co-pyrolysis process), the co-pyrolysis chars showed that the rubber char mixed well with the sawdust char and obtained higher specific surface area values by forming more expanded porous structure.

As shown in Fig. 6a, the untreated rubber char presented a high crystallinity of $CaCO_3$ phase, which was reasoned that the functional fillers improved the mechanical and biodegradable properties of rubber (Jin and Park 2008). The caking char was observed in Fig.5, (Rubber-A). The diffuse X–ray peaks in the range of 10–30° demonstrated that the amorphous carbon structure was initially formed in the untreated char of sawdust during the graphite process of sawdust pyrolysis (Lin et al. 2011). After the demineralization, most of $CaCO_3$ in rubber was transformed into $CaCl_2$ as observed in Fig. 6b. It was corresponding to the decrease of ash content as demonstrated in Table 2. Fig. 6c shows that the $CaCO_3$ was almost eliminated and a diffraction peak of K_2O phase (at 32.8°) was observed after the activation. It was illustrated that the chemical reaction at a high temperature occurred between KOH activator and carbons in char, which generated much small molecule gas such as CO and H_2, resulting in promoting the formation of pore structure (Lillo-Rodenas et al. 2003). It was consistent with many micro-pores in the co-pyrolysis chars observed in Fig. 5 (1:1-B).

CONCLUSIONS

The low valued rubber and sawdust were converted into value-added activated carbon through the two-step co-pyrolysis (pyrolysis and activation). The co-pyrolysis char produced by adding rubber in sawdust obtained low activated char yield but higher BET surface area values. Upgrading procedures efficiently extracted the residue oil and reduced ash content to obtain much cleaner carbon. During activation process, the chemical reaction at a high temperature occurred between KOH activator and carbons in char, which generated much small molecule gas such as CO and H_2, resulting in developing better porous structure and higher BET surface area values.

ACKNOWLEDGEMENT

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INFLUENCE OF ULTRASOUND-ASSISTED EXTRACTION ON VOLATILES AND RESIDUES IN WOOD PYROLYSIS

Zhenyu WANG
College of Material Science and Technology, Beijing Forestry University, No.35 Tsinghua East Road Haidian District, Beijing, PR China
E-mail: wangzhenyu1992@126.com

Lijie QU
College of Material Science and Technology, Beijing Forestry University, No.35 Tsinghua East Road Haidian District, Beijing, PR China
E-mail: QLJie2016@bjfu.edu.cn

Jiali ZHANG
College of Material Science and Technology, Beijing Forestry University, No.35 Tsinghua East Road Haidian District, Beijing, PR China
E-mail: zjl0520@yeah.net

Zhengbin HE
College of Material Science and Technology, Beijing Forestry University, No.35 Tsinghua East Road Haidian District, Beijing, PR China
E-mail: hzbcailiao@bjfu.edu.cn

Songlin YI
College of Material Science and Technology, Beijing Forestry University, No.35 Tsinghua East Road Haidian District, Beijing, PR China
E-mail: ysonglin@126.com

Jun MU
College of Material Science and Technology, Beijing Forestry University, No.35 Tsinghua East Road Haidian District, Beijing, PR China
E-mail: mujun222@sina.com

Abstract

Eucalyptus was used as the raw material for treatment with ultrasound-assisted extraction (UAE) to study the ultrasonic effects on volatiles emitted and residues produced during pyrolysis. A swept ultrasonic cleaner with a frequency of 40 kHz and an intensity of 360 W was applied to samples at 60°C for 30 minutes. The characteristics of the ultrasound-treated samples were then compared with those of Soxhlet-extracted and untreated biomass by TG-FTIR and SEM. The results showed both mechanical and chemical effects of ultrasound played a significant role in efficiently altering biomass characteristics. In thermogravimetric analysis, UAE samples showed the highest maximum mass loss rate (-52.1%/min) with the lowest temperature (378.4 °C), while the other two samples presented similar trends to each other with lower maximum mass loss rate. Volatile profiles obtained by TG-FTIR indicated that CO and methanol components were mainly influenced by extraction, while CO2, CH4, and formic acid responded more strongly to the effects of ultrasound. After UAE, an increase in CO, CH4 and decreases in CO2 and formic acid were produced during pyrolysis. From SEM images, the fracture of pit membranes clearly showed the mechanical effects of ultrasound, which accounted for the significant enhancement of extraction of valuable ingredients.

Key words: *pyrolysis; TG-FTIR; ultrasound; volatile evolution; wood.*

INTRODUCTION

The rapid consumption of fossil fuels and environmental degradation have prompted researchers to investigate on alternative energy. Renewable biomass reduces reliance on fossil fuels and it does not add new carbon dioxide to the atmosphere (Kendry 2002). As a result, renewable biomass has been regarded as one of the most appropriate substitutes in recent decades. In addition, the exploiting and utilization of renewable biomass contribute to the sustainable development of society. Eucalyptus, a fast-growing species, is currently widely-planted in many different parts of the world. For example, approximately 3.5 million hectares of eucalyptus plantations, which can produce

15-30 m3/ha/a have been established in Southern China alone (Zhou and Wingfield 2011). At present, a majority of the eucalyptus resources are used for pulp production (Oudia et al. 2009) and furniture making, which itself produces a large amount of wood residue and generates lower profits. Consequently, making full use of eucalyptus resources by biomass conversion can not only address energy crisis and increase the value of biomass, but also greatly mitigate the environmental burdens caused by biomass waste (Părpăriţă et al. 2014).

Among methods of biomass conversion (pyrolysis, combustion, fermentation, etc.), thermochemical technologies have attracted increasing interest due to the flexibility and simplicity of conversion process (Yanik et al. 2007). Pyrolysis is a fundamental thermochemical conversion process that can be used to transform biomass directly into gaseous and liquid fuels. And pyrolysis is also an essential step in combustion and gasification processes (White et al. 2011).

Additionally, in order to take full advantage of biomass, diversified methods can be applied prior to pyrolysis process, particularly ultrasound-assisted extraction (UAE) for valuable components. For many years, ultrasound was used to increase the yield of extractives from biomass with reduced processing time, mainly due to the effect of cavitation. Cavitation refers to the formation, growth, and violent collapse of cavities caused by ultrasound in the liquid (Sutkar and Gogate 2009), which can efficiently improve the penetrability of biomass and intensify the mass transfer significantly (Toma et al. 1999; Vinatoru 2001). A number of researchers have studied the utilization of UAE, aiming to produce natural products and chemicals with high utility value in cosmetics, medicines, and other consumer goods (Shirsath et al. 2012). Li et al. (Li et al. 2004) extracted oil from two varieties of soybeans with the application of 20 kHz high-intensity ultrasound and found that high-intensity ultrasound reduced the time required to extract edible oils from plant sources, consequently improving throughput in commercial oil production processes. The extraction of anthocyanins from red raspberries by an ultrasound-assisted process was studied by Chen (Chen et al. 2007), and the optimized conditions was obtained at 22 kHz frequency.

Accordingly, UAE can be used as an appropriate treatment prior to pyrolysis for higher value applications of biomass. Widely-used as the ultrasonic treatment is, to the best of our knowledge, the studies focusing on the characteristics of pyrolysis of ultrasound-treated biomass are insufficient. Guo et al. (Guo et al. 2010) investigated the differences between untreated biomass and extracted residues in pyrolysis and reported that the extractives facilitate the formation of acetic acid and inhibit the formation of levoglucosan. Wang et al. (Wang et al. 2011) indicated that extractives enhance not only oil yield, but also alkane content during pyrolysis. However, such studies could merely reflect the extraction effects of ultrasound, and they didn't take into account the chemical effects caused by ultrasound. Furthermore, although many existing studies had demonstrated morphologic changes in samples after ultrasonic treatment (Chen et al. 2011), the structural changes of treated pyrolysis residues were not characterized. More importantly, since volatiles are one of the most valuable products and energy substance in pyrolysis, the lake of studies about the influence of ultrasound on volatiles released during pyrolysis also need addressing. Consequently, the purpose of this study is to explain the various effects that UAE exerts on biomass pyrolysis, especially on the release process of volatiles and morphologic characteristics of the pyrolysis residues.

MATERIAL, METHOD, EQUIPMENT

2.1. Raw materials

The Eucalyptus (Eucalyptus grandis × Eucalyptus urophylla) used for this study was provided by Guangxi Ushine Home Products Limited Company in China. Chemicals were purchased from Beijing Chemical Works. Raw materials were ground in a grinder (type XTP-1000A, Zhejiang Red Sun Machinery Co., Ltd, China) and sieved to a particle size below 0.45mm. Samples of three different treatment conditions were analyzed: (1) untreated biomass, (2) Soxhlet-extracted biomass, and (3) ultrasound-extracted biomass. All samples were dried at 80°C for 24 hours to a constant weight prior to treatment and test. The proximate and ultimate analyses of samples were carried out by a muffle furnace and an Elementar Vario EL III Analyzer (Elementar Analysensysteme GmbH, Germany) respectively. And the results were presented in Table 1.

Table 1

Proximate and ultimate analyses of different samples

samples		Untreated sample	SE sample	UAE sample
Ultimate analysis (wt%,db)	C	49.86	49.69	49.39
	H	5.63	6.00	5.99
	O	43.96	44.15	44.63
	N	0.26	0.22	0.18
Proximate analysis (wt%,db)	Ash	0.24	0.15	0.12
	Volatile matter	86.83	87.38	88.04
	Fixed carbon	12.93	12.47	11.84

db: dry basis.

2.2. Extraction

Samples undergoing extraction were extracted by either Soxhlet or ultrasound method. Each extraction sample contained 3g of untreated biomass and 150ml of solvent, a 2:1 (V/V) mixture of benzene and ethanol. During the process of extraction, a Soxhlet apparatus operating at 90°C for 6h, or conversely, a bath-type ultrasonic device for indirect UAE was used to obtain extractives from raw materials. Though several factors influence UAE results (Wang and Weller 2006), exploring the optimal extraction conditions was not the purpose of this study; the parameters of ultrasonic extraction were determined according to a preliminary experiment. UAE was conducted in a conical flask immersed in the middle of a SB-400DTY Swept Ultrasonic Cleaner (Suzhou Jiangdong Precision Instrument CO., Ltd, China) with a frequency of 40 kHz and an intensity of 360 W at 60°C for 30 minutes, which was enough to bring about complete extraction.

2.3. TG-FTIR

The process of volatile emission was examined using a Netzsch STA449F3 computerized thermogravimetric analyzer (TGA, NETZSCH Gerätebau GmbH, Germany) combined with Fourier transform infrared spectroscopy (FTIR). Approximately 20mg of samples were heated to 900°C at a heating rate of 50°C/min. Purified argon was introduced into the reactor at a steady flow rate of 20 ml/min to create an oxygen-free atmosphere and immediately remove the gaseous and condensable products to avoid secondary interactions. The argon flow swept products into the gas cell of the FTIR spectrometer (Bruker Vertex 70v, Bruker Corporation, Germany) with a resolution of 4 cm-1 and five scans per measurement. The IR scanning range was set from 4000 to 500 cm-1. This system enabled monitoring of the time-dependent evolution of volatiles and the weight of the pyrolysis residues. Interpretation of FTIR spectra was made according to the NIST spectra database and empirical literature (Koutsianitis et al. 2015; Linstrom and Mallard 2016; PăRpăRiţĂ et al. 2014; Yang et al. 2007; Zhang et al. 2016).

2.4. Scanning electron microscopy (SEM)

The microscopic structures of pyrolysis residues extracted by the Soxhlet and ultrasound methods were compared to that of the untreated biomass using scanning electron microscopy (SEM, Hitachi S-3400N II, Japan). Prior to analysis, the samples with particle size of 0.3-0.45 mm were sputter-coated with gold. These samples were then analyzed under the high vacuum at an operating voltage of 5.00kV.

RESULTS AND DISCUSSION

3.1. Thermogravimetric analysis

The pyrolysis process of all samples were recorded by thermogravimetric analyzer and shown in Fig. 1. The TG/DTG curves showed that all three samples began rapid decomposition at around 240°C, before which a slight mass loss can be observed in the extracted biomass. Except for moisture volatilization of raw materials occurring below 100°C, the mass loss of the extracted biomass in low temperature (100-200°C) might result from the dehydration and depolymerization of unsteady hemicellulose, which was enhanced by pre-extraction. In the DTG curves, there were two dominant peaks appearing in all samples during pyrolysis. As the data reported by Antal and Varhegyi showed (Antal and Varhegyi 1995), the low-temperature peak was connected to the decomposition of hemicellulose, the high-temperature peak was due to cellulose decomposition, and lignin decomposed over a broad temperature range. In Fig. 1, the main decomposition stage continued to approximately 500°C and then the flattened TG/DTG curves appeared, due to aromatic condensation (Brebu et al.

2013). At the end of the measurement, the TG curves suggested that there was less residues for extracted biomass after pyrolysis. Although the amount of residue was similar, the UAE's shorter time scale than the Soxhlet extraction (SE) showed the high-efficiency of ultrasound. After extraction, the thermal stability of samples decreased as compared to that of the untreated biomass, but there were differences between the UAE and SE groups. The maximum rate of degradation of the three samples occurred at 378-382°C in the DTG curves, the lowest temperature being observed for the ultrasound-extracted biomass, which also possessed a higher maximum rate of degradation than the others.

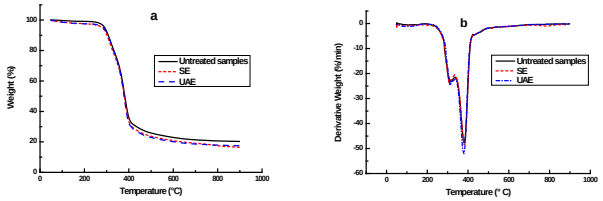

Fig. 1.
TG(a) and DTG(b) curves of the three samples at a heating rate of 50°C/min.

3.2. FTIR spectra of volatiles emitted during pyrolysis

In order to investigate the mass loss behavior shown in the TG and DTG curves, an FTIR spectrometer was applied to analyze the volatiles emitted during the pyrolysis. The FTIR analysis of volatiles from the TG device shows bands of functional groups, although it is difficult to identify particular compounds due to signal overlap, especially for complex mixtures. However, specific components such as H2O, CO2, CH4, etc., may be inferred. The infrared absorption band assignment corresponding to the main volatiles from pyrolysis is shown in Table 2.

Table 2

Infrared absorption band assignment for volatiles from biomass pyrolysis

Wavenumber,cm^{-1}	Infrared absorption band assignment	Products
3950-3500	O–H stretching vibration	H_2O
1900-1300	H–O–H stretching vibration	
3014	$–CH_3$ stretching vibration	CH_4
3200-2850	C–H stretching vibration	
2360, 669	C=O stretching vibration	CO_2
2177, 2114	C–O stretching vibration	CO
1850-1700	C=O stretching vibration	Aldehydes, ketones
1601,1509	aromatic rings	
1400-1300	O–H stretching vibration	Alcohols, phenols

The FTIR spectra of all biomass volatiles at the maximum volatile release are shown in Fig. 2. In accordance with the widely-used Lambert-Beer law, the absorption spectrum at a specific wavenumber is linearly correlated with the concentration of specific volatiles (Bassilakis et al. 2002). In general, the biomass yielded non-condensable gaseous products consisting mainly of CO2 (2360, 669 cm-1), CO (2177, 2114 cm-1), and CH4 (3014 cm-1). Other condensable volatiles of significance included H2O, aldehydes, carboxylic acids, ketones and alcohols. The spectra indicated that the species of volatiles emitted from different samples were almost identical, because the adsorption bands appeared to be at nearly the same wavenumber. However, there were diversities in the intensity of absorption peaks, which suggested differences in the quantity of volatiles. Compared to the other two samples, biomass treated with ultrasound showed several stronger absorption peaks, which were consistent with the higher mass loss rate in Fig. 1.

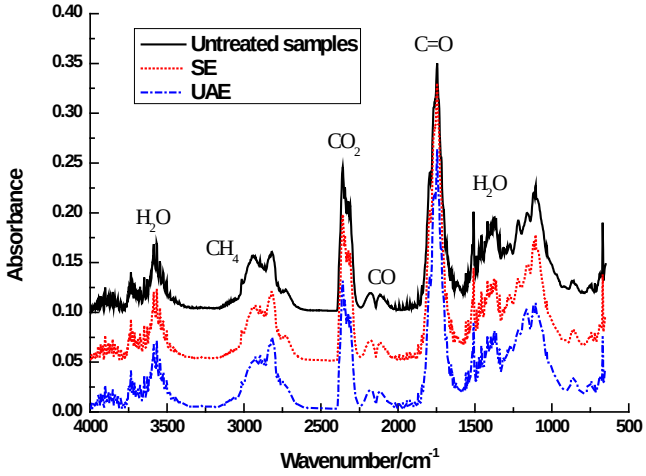

Fig. 2.
FTIR spectra of pyrolysis volatile products for all samples at the maximum mass loss rate.

Based on the existence of characteristic bands at 3500-3950 cm-1 and 1300-1900 cm-1, the release of water was observed at this stage. The wide spectral regions at 2850-3200 cm-1 showed the existence of C–H stretching vibration identifying the volatile species together with other characteristic bands, such as formic acid (2943, 1770, 1105 cm-1), acetic acid (2944, 1791, 1182 cm-1), and acetone (2937,1731,1216 cm-1). The bands at 2710-3140, 1300-1400, 960-1085 cm-1, were characteristic of one of the important alcohols, methanol, which released continuously in a significant amount. Phenols derived from lignin polymer were also crucial products in biomass pyrolysis, and these phenols correspond to absorption bands at 1300-1400 cm-1. However, the species of phenols were difficult to confirm in Fig. 2, due to the signal overlap. In addition, the absorption bands of formaldehyde at 2770-2860, 1746, 1508, and 1167 cm-1, levoglucosan at 1183 cm-1, and hydroxyacetaldehyde at 860 cm-1 all indicated their formation in the temperature range of maximum volatile release (Li et al. 2001; Liu et al. 2009).

3.3. Releasing property of volatiles from biomass pyrolysis

Figs. 3(a-c) display typical stack plots of FTIR spectra from biomass pyrolysis using TG-FTIR. The FTIR spectra taken from 50 to 900°C were plotted one on top of the other to form the 3D spectra. These plots indicated the evolution of volatile products during pyrolysis, as a function of both wavenumber and temperature (Yang et al. 2007). More accurate identification of compounds was possible by analyzing multiple signals, because the temperature evolution of some compounds were different (Brebu et al. 2013). In addition, more complete distribution of absorption bands could be presented with the help of 3D spectra. For example, the bands at 1714, 1267, 1004 and 750 cm-1 in the 3D spectra represented the formation of furfural, which did not clearly appear on the spectra corresponding to maximum volatile release. Furthermore, the maximum volatile release of the three samples occurred at 388-393°C in the stack plots, while the maximum rate of degradation was at 378-382°C in the DTG curves. These small differences in characteristic temperatures of pyrolysis determined from FTIR and DTG data were expected, due to transportation of the volatile products from the TG furnace to the FTIR detectors through the transfer lines.

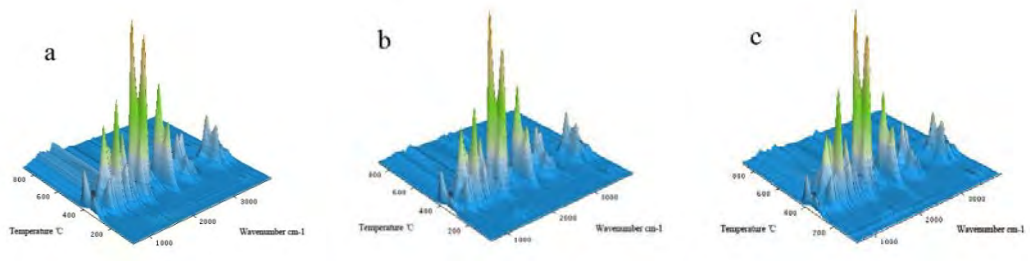

Fig. 3.
Typical stack plots of FTIR spectra from the three pyrolysis samples: (a) untreated samples; (b) Soxhlet extracted samples; (c) ultrasound-extracted samples.

Fig. 4 shows the specific FTIR profiles of several products evolving from biomass pyrolysis, including CO2, CO, CH4 and several organics. Based on the Lambert-Beer law, the variation of absorbance across the entire pyrolysis process reflected the concentration trend of the volatile species (Gao et al. 2013). Thus, these profiles described the release process of volatiles. It was found that the majority of initiation reactions became significant above 200°C, and almost all of the volatiles presented two dominant peaks in the neighborhood of 320°C and 390°C. The high-temperature peaks of the ultrasound-treated samples were shifted slightly lower, which was consistent with the two distinct mass loss peaks in the DTG curves in Fig. 1.

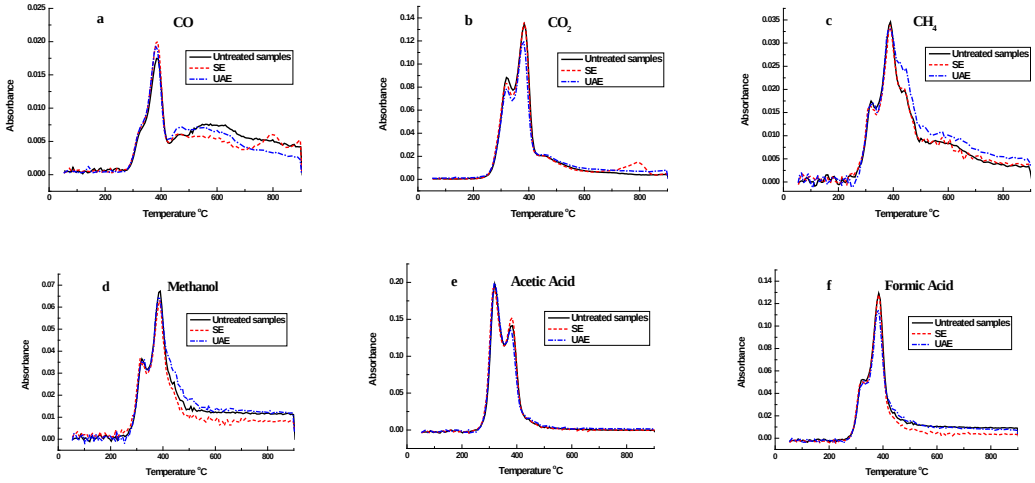

Fig. 4.
FTIR profiles of volatiles evolving from the biomass pyrolysis.

Absorption peaks at 2177 and 2360 cm-1 resulting from the C–O stretching vibration and C=O stretching vibration, respectively, were used to construct the formation profiles of CO2 and CO. CO was a decomposition product of ether groups, and originates from the ether bridges of lignin subunit connections and/or the ether compounds in the secondary cracking of volatiles (Zhang et al. 2016). Additionally, cellulose pyrolysis and the break of lateral chains in lignin polymer, such as the aliphatic hydroxyl group and the C–C bond also generated CO (Fenner and Lephardt 1981). By comparison, hemicellulose contributed less to the amount of CO produced in pyrolysis, so that, compared to the other products, the low-temperature peak (~320°C) of CO in Fig. 4(a) was not apparent. At high temperature (>500°C) the increase of CO was most likely to be attributed to the secondary pyrolysis of tar residue in the solid sample. The secondary reactions can be expressed as follows:

$$C + H_2O = H_2 + CO \quad (1)$$

$$C + CO_2 = 2CO \quad (2)$$

As discussed by Yang (Yang et al. 2007), the releasing of CO2 was mainly caused by the cracking and reforming of functional groups of carboxyl and carbonyl in hemicellulose under 500°C. Lignin contributed a considerable amount of CO2, most notably by decarboxylation at around 400°C (Jakab et al. 1995), whereas cellulose resulted in a small amount of CO2, due to the lowest content of C=O group in biomass. For cellulose, possible mechanisms to produce CO2 involved depolymerization reactions of the active cellulose and decomposition of levoglucosan to volatiles with low molecular weight (Banyasz et al. 2001; Kawamoto et al. 2003). The strongest peak of CO2 occurring at ~390°C can be regarded as the combined action of cellulose and lignin.

In Fig. 4(c), the absorption peaks at 3004 cm-1 were extracted to plot the releasing profiles of methane, whose evolution occurred at low levels throughout the pyrolysis process. However, sharp rises in methane formation were observed between 300-500°C. At temperatures above 250°C, radical reactions that played vital roles in describing degradation of lignocellulosic structural material became significant. CH3 and H radicals formed from H abstraction and β-carbon scission reactions could begin to form CH4 (Butterman and Castaldi 2007). As the temperature increased, sharp peaks appeared near 400°C, while broad peaks formed in the high temperature range of 500-600°C. CH4 released at lower temperature mainly came from the decomposition of methyl and methoxy functional groups, while the decomposition of the hydrocarbon skeleton after deoxygenation, as well as the following molecular methanation reactions, can partly account for CH4 produced above 400°C (Butterman and Castaldi 2007; Faix et al. 1988; Jakab et al. 1995).

$$2CO + 2H_2 = CH_4 + CO_2 \quad (3)$$

$$C+CO_2= 2CO \quad (4)$$

$$C + 2H_2 = CH_4 \quad (5)$$

Alcohols and phenols were identified by the significant bands of -OH at 1300-1400 cm-1. Furthermore, methanol, one of the most important alcohols, was observed, based on the characteristic bands at 2710-3140 and 960-1085 cm-1. From the study of Jensen et al. (Jensen et al. 1998), methanol was not formed in the pyrolysis of cellulose, and therefore the profiles of methanol in Fig. 4(d) originated primarily from the decomposition of hemicellulose and lignin. Although there are large amounts of hydroxyl in hemicellulose, alcohols were not superior in numbers as compared to other organics. The oxidization of hydroxyl during pyrolysis might account for this result, due to the transfer of hydroxyl into carboxyl and carbonyl. As the temperature increased, the cracking of C-C bonds in xylose could facilitate the formation of alcohols. In terms of lignin, the methoxyl substituents were the most probable contributors to the formation of methanol (Liu et al. 2008). Furthermore, free hydroxyl groups generated by the breakage of lignin structure at lower temperature could reform with C-O bonds subsequently forming methanol.

Figs. 4(e, f) show the profiles of acetic acid and formic acid at 1791 cm-1 and 1105 cm-1, respectively. The obvious discrepancies in volatile evolution between acetic acid and formic acid reflect the different mechanisms of acid formation. Based on existing studies, acids produced in biomass pyrolysis were mostly contributed by the degradation of hemicellulose and cellulose, while those from lignin were negligible (Yang et al. 2007). For acetic acid, peaks were higher at lower temperature (~320°C) than those near 390°C, which suggested that it was mainly derived from hemicellulose. And specifically, studies showed acetic acid could be related to acetyl groups in the main xylan chain (Ponder and Richards, 1991; Shafizadeh et al. 1972). Compared with acetic acid that was apt to present the maximum volatile peak at lower temperature, the peaks of formic acid in the range of 300-400°C were not completely separate and a higher temperature was required for the generation of formic acid.

From Fig. 4, the effects of ultrasonic treatment on the pyrolysis of biomass are shown. It was found that extraction played a major role in the formation of CO and methanol, because the changes in both UAE and SE groups were obvious when compared with untreated samples. After extraction, more CO and less methanol were released at the dominant peak around 390 °C. CO produced after UAE was less than that generated in SE group, likely due to the ether bonds in lignin cleaved by ultrasound (Yoshioka et al. 2000). Figs. 4(b, c, f) imply that the ultrasonic effects were closely connected with the formation of CO2, CH4 and formic acid. Under the influence of ultrasound, less CO2 and formic acid were emitted during the degradation process, while more CH4 was detected especially at the temperature range above 400°C. Moreover, the acetic acid profiles of all samples are similar to each other, except for small differences in the high-temperature peak, which may result from the joint effects of ultrasound and extraction.

3.4. Scanning electron microscopy (SEM)

According to the TG curves in Fig. 1(a), the amount of residue after pyrolysis was represented. Among the three samples, the untreated biomass possessed the highest residue content (~20.3%), with the other two samples showing similar values at around 17.6%. SEM images were used to observe the surface morphology of both the untreated and extracted residues. The SEM analysis results are shown in Fig. 5 both at a low magnification of 2.00k (Figs. 5(a-c)) and a high magnification of 4.00k (Figs. 5 (d-f)). From the low magnification micrographs, the biomass residue appeared irregular and the shape significantly altered after pyrolysis. Due to the thermal effects of pyrolysis, all samples became thin and twisted. In Figs. 5(d-f), these residues still showed at least partial retention of the initial structure of the walls and pits at the higher magnification micrographs. Pit membranes were broken up in the group treated with ultrasound, while they were nearly integrated in the untreated samples. The destruction of pit membranes could be ascribed to the ultrasonic cavitation that exposed the cell surface to fluid jets, creating mass transfer channels which significantly improved the penetrability of the biomass. Furthermore, compared with other samples, multiple small protuberances were observed at the surface of residues treated by ultrasound, which was likely to imply much deeper changes to the biomass structure, such as the melt and repolymerization of lignin (Shen et al. 2016). This peculiar surface structure may lead to further reaction and contribute to more volatiles, as suggested in Fig. 2 and 3.

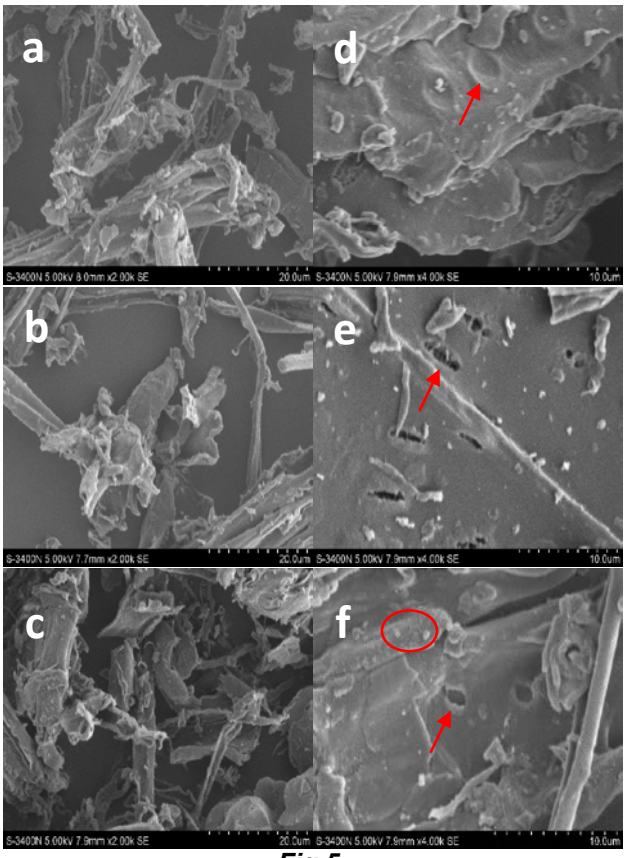

Fig 5.
Scanning electron micrographs of pyrolysis residues at different magnifications (2.00k and 4.00k): (a) and (d) untreated samples; (b) and (e) Soxhlet extracted samples; (c) and (f) ultrasound-extracted samples.

3.5. Influence mechanism of ultrasound on wood pyrolysis

According to the literature (Ebringerová and Hromádková 2002; Shirsath et al. 2012; Sun and Tomkinson 2002), ultrasonic treatment impacts raw materials mainly due to mechanical and chemical effects caused by cavitation. Because of the propagation of ultrasound pressure waves through the solvent, the cavities collapse, leading to generation of localized ''hot spots'' with transient temperatures of the order of 10,000K and pressures of about 1000 atm (Saharan et al. 2011). In addition, cavitation also results in strong liquid circulation, high shear stress near the bubble wall, and formation of micro-jets near the solid surface (Mason and Lorimer 2003; Sutkar and Gogate 2009). Consequently, mechanical effects appeared to cause disruption of cell walls, particle size reduction, and enhanced mass transfer across cell membranes in the macroscopic view. In the microscopic view, chemical effects were primarily the generation of highly-reactive free radicals and cleaves of the inter- and intra-molecular linkage of the main wood composition (Sun et al. 2004; Sutkar and Gogate, 2009). The effectiveness of chemical effects is closely related to the solvent used in the experiment. For example, water molecules can dissociate into OH· and H· under conditions of high pressure and temperature generated by ultrasound cavitation (Badve et al. 2014). And the radicals produced in ultrasonic treatment lead to a series of reactions in biomass. However, the generation and influence of radicals are not clear when a solvent comprising of benzene and ethanol is used.

From the results presented in this study, it was both mechanical and chemical effects that contributed to changes in the characteristics of biomass pyrolysis, especially in volatile evolution. And the mechanical effects often outweighed chemical effects to some degree. Mechanical effects ultimately resulted in the removal of extractives, which greatly impacted pyrolysis, just as what classical extraction does. Additionally, the important aspects of UAE lay in the enhancement of extraction efficiency which was demonstrated by the shorter extraction time and lower extraction temperature used in the study, as well as the damaged structure seen in SEM images. In comparison, chemical effects mainly break down the chains and linkages between wood components, leading to decreases in molecular weight and thermal stability (Sun et al. 2004). Although the changes were not readily apparent, chemical effects did produce noticeable differences between UAE and SE samples, particularly in the mass loss rate and the process of volatile emission. These less-obvious results also

agreed with studies claiming that ultrasound mainly acts on branched structure of wood components, but without significant destruction of the primary structural features (García et al. 2011; Sun et al. 2002a; Sun et al. 2002b).

CONCLUSIONS

In the present work, UAE and SE were applied to eucalyptus in organic solvent. The pyrolysis characteristics, as well as volatiles emitted and residues produced in pyrolysis, were the primary focus of this research. Resulting TG curves showed that samples that had undergone UAE and SE presented comparable residues (~17.6%), but the amount was less than that for the untreated samples. As shown in the DTG curves, UAE possessed the highest maximum mass loss rate (-52.1%/min) with the lowest temperature (378.4 ° C), while the curves of the other two samples are similar. With the utilization of TG-FTIR, the process of volatile emission was also explored. The results indicated that CO and methanol were mainly influenced by extraction, while CO2, CH4, and formic acid responded to the effects of ultrasound. Following UAE, more CO, CH4 and less CO2 and formic acid were produced during pyrolysis, which benefited the utilization of biomass energy. SEM images indicated partial retention of the biomass structure, and the fracture of pit membranes clearly showed the mechanical effects of ultrasound on the samples.

By this study of ultrasound-assisted extraction and wood pyrolysis, UAE, an environmental-friendly and high-efficiency process for the recovery of valuable components from biomass, is expected to be used in an extensive range and make full use of biomass resources in the combination with pyrolysis.

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FUEL PROPERTIES OF OAK, POPLAR AND PINE LOGGING RESIDUES

Vasilis FILIPPOU
Laboratory of Forest Products Technology, Aristotle University of Thessaloniki University Campus
54124 Thessaloniki Greece, E-mail: vafilpo@teilar.gr

Ioannis PHILIPPOU
Laboratory of Forest Products Technology, Aristotle University of Thessaloniki University Campus
54124 Thessaloniki Greece, E-mail: jfilippo@for.auth.gr

Ioannis ELEYTHERIADHS
Centre for Renewable Energy Sources and savings (CRES)
19009, Athens, Greece, E-mail: joel@cres.gr

Konstantinos TSIOTAS
Centre for Renewable Energy Sources and savings (CRES)
19009, Athens, Greece, E-mail: ktsiotas@cres.gr

Abstract

The growing global energy demand and concerns about the negative effects of growing greenhouse gas (GHG) emissions from fossil fuels call for alternative energy sources. One such renewable resource is logging residues that remain in the forest after harvesting. Exploring the possibilities of utilizing the biomass of logging residues for energy requires analysis and knowledge of its properties. In this research work the properties (percentage of bark, ash, volatiles, fixed carbon, carbon, hydrogen, oxygen, nitrogen and calorific value) of the various constituents of the biomass of oak (Quercus frainetto), poplar (Populus alba) and pine (Pinus nigra) logging residues were determined. Bark and ash content increased with decreasing diameter of branches. Ash content was higher in bark than in wood of branches in all species. Ash content of the all thick and thin branches was in oak 2,53% and 3,81%, in poplar 1,12% and 1,58% and in pine 0,79% and 1,16%, respectively. Ash content of twigs was in oak 4,14% and in pine 2,27%. Nitrogen content of branches varied from 0,105% to 0,312% and it was higher in oak and in thin branches. N content of twigs was 1,173% in oak and 0,76% in pine. Oak branches and oak and pine twigs had ash and nitrogen content higher than that required by the EN ISO 17225-2 standard for domestic pellets and they should not be used for energy, at least for pellets production. Volatile mater, fixed carbon, carbon and hydrogen content were in the range given by other researchers. Heating value ranged between 18,27MJ/kg to 21,0% and it was higher in pine than in oak and poplar, and higher in twigs and thin branches.

Key words: *forest biomass; logging residues; oak; poplar; pine; energy properties.*

INTRODUCTION

The growing global energy demand and concerns about the negative effects of growing greenhouse gas (GHG) emissions from fossil fuels call for alternative energy sources, which are low cost, renewable and non-polluting. One such renewable resource is biomass, especially forest biomass (European Commission 2005, Smeets and Faaij 2007, Ladanai and Vinterbäck 2009, Becker et al. 2011).

In recent years, the use of residues that remain in the forests after logging has attracted great interest as an energy source (Lehtikangas 2001, Gan and Smith 2006, Gan and Smith 2007, Nurmi 2007, Eker et al. 2009, Hu and Heitman 2008, Malinen et al. 2010, Giuntoli et al. 2015, Filippou et al. 2015, Roser et al. 2008, Philippou 2014).

The biomass consists of tops, branches, bark, foliage or needles and stumps. Forest residues may also include small trees that break during logging, dead trees and low-value trees or trees of non-market forest species (Roser et al. 2008, Philippou 2014).

In the past, logging residues were not exploited mainly because their harvest and transport was technically difficult and uneconomic. Currently new harvesting technologies and transportation systems have been developed and in conjunction with the increase in petroleum prices enable their extraction from the forest (Kauriinoja 2010, Svanaes and Jungmeier 2010, Filippou and Philippou 2014). Also, new and more efficient technologies enable conversion of biomass into energy in small units (mainly gasification) or conversion into compressed forms (wood pellets) that can be installed in

or near the forests (Filippou and Philippou 2014). These further limit transportation costs and give opportunities for local employment and rural development. Thus, logging residues from final harvest are expected to play an important role in meeting renewable energy goals in many countries (Gan and Smith 2006, Nurmi 1993). Their utilization for energy could create business opportunities and employments in local populations, generate profit from residual material and provide energy self-sufficiency for rural communities (Aguilar 2014).

Compared with the usual stem wood, biomass of logging residues differ in chemical composition % of cellulose, hemicelluloses, lignin, extracts and inorganic elements (Nordin 1994, Nurmi 1993, Nurmi 1997, Zeng 2014). There also exists variability in chemical composition between the various constituents of forest biomass (Philippou 1982, Werkelin et al. 2005, Wang and Dibdiakova. 2014). Moisture content, ash content, volatile content, elemental composition and calorific value are the main material properties that affect the material behavior during conversion into energy as well the overall energy outcome (Obernberger et al. 2006, Vassilev et al. 2010).

The ash content of biomass is known to vary between tree species and tree components (Hytonen and Nurmi 2015, Rhén 2004). High ash content can decrease the heating value of biomass. In addition, ash content and its composition affect the proper functioning of the burners and gassifiers (Bryers 1996, Raask 1969). The ash adheres to the heat transfer surfaces and cause corrosion. When burning the elements, mainly K, Na, S and Ca can melt, form sticky particles, adhere to the surfaces of the walls and create a burner malfunction (Raask 1969). The biofuel content of nitrogen N is responsible for the formation of NOx which have an environmental impact (Munalula and Meincken 2009). For biomass pellets, there is a need to have a low ash and nitrogen content in order to meet quality standards requirements (Filbakk et al. 2011, EN ISO 17225-2:2014).

Calorific value of biomass is a function of its chemical composition. Various researchers have determined the calorific value of various types of biomass from their elemental composition using proximity regression analysis models (Demirbas 2003, Friedl et al. 2005, Telmo et al. 2010, Singh et al. 2015). Several researchers (Nurmi 1997, Zeng et al. 2014, Philippou 1982, Obernberger et al. 2006, Harris 1984, Howard 1973, Howard 1988, Demirbas 1997) have measured the heating value of various tree species and various tree components and found significant differences both between species and between tree biomass components.

Proper utilization of logging residues for energy requires analysis and good knowledge of their properties. The aim of this work was to look at the branches of oak, poplar and pine that remain in the forest after harvesting and determine their properties that affect energy efficiency. The properties studied included percentage of bark, moisture, ash, volatiles, fixed carbon, carbon, hydrogen, oxygen, nitrogen and calorific value.

MATERIAL AND METHODS

Representative samples of oak (*Quercus frainetto*), poplar (*Populus alba*) and pine (*Pinus nigra*) branches with bark and foliage were taken from five trees of each species from a mixed forest in northern Greece during normal logging operations. For determining the % of bark in branches transverse discs of different diameters (from 2 to 9cm) were cut (Fig. 1). The percentage (%) of bark was calculated by measuring the diameter of the disc with the bark and after peeling the bark using the formula:

(1)

$$\text{bark } \% = \frac{d_1 - d_2}{d_1} \times 100$$

where:
d_1=disk diameter with bark
$d_{2=}$ disk diameter without bark

Fig. 1.
Disks of branches of various diameters for measuring % of bark.

The branch samples were in total 170 and had a diameter of 2cm to 9cm. Measurements were carried out in 4 discs (repetitions) of each branch. Regression analysis was used to find any relationships between branch diameter and bark percentage.

For the determination of other properties, the branches were cut into three parts: thick branches (diameter >5cm), thin branches (diameter of 2 - ≤5cm) and twigs (branches with a diameter <2cm including the needles or leaves). Samples of thick and thin branches were debarked in order wood and bark to be tested separately. The samples were air-dried and milled first in a common hammer mill and then in a Willey mill to obtain particles having a size <0,420mm (40 mesh). Ash content (% dry weight), the percentage of volatiles and the fixed carbon, and the elemental analysis (C, H, N) were determined accordance with CEN/TS 14775 (2005), CEN/TS 15148 (2005) and CEN/TS 15104 (2005) standards, respectively. The higher heating value (MJ/Kg dry) were determined in accordance with CEN/TS 14918 (2005) standard. Three samples of each material were used for the measurements of each property.

RESULTS AND DISCUSION

Percentage (%) bark

Table 1 shows the average bark percentages of all branches measured as well as the average percentage in each of the two class sizes of branches of oak, poplar and pine. Bark % was much higher in oak than in pine and poplar and increased with decreasing branch diameter. The differences in the percentage of bark between species was more evident when it was calculated at the same branch diameter (d = 5cm) for the three species. Table 2 and Fig. 2 give the regression analysis models of the effect of branch diameter on bark percentage.

Table 1

Bark percentage (%) of branches

Species	Branches						
	d=2-9cm		d=2-5cm		d=>5cm		d=5cm
	đ* a/a**	% bark	đ a/a	% bark	đ a/a	% bark	% bark++
Oak	5,86* 32**	6,89 (1,32)+	4,02 16	7,85 (1,01)	7,97 16	5,85 (0,52)	7,30
Poplar	5,93 35	3,98 (0,79)	3,92 19	4,46 (0,54)	8,02 16	3,04 (0,39)	4,19
Pine	5,91 32	4,09 (0,71)	4,05 17	4,52 (0,83)	7,97 15	3,61 (0,55)	4,49

* Average diameter, **No of samples, +standard deviation, ++ calculated

Table 2

Correlation models with the best fit between branch diameter and percentage of bark

Species	Mondel	R^2
Oak	$y = -0,055x^2 + 0,189x + 8,065$	0,794
Poplar	$y = -0,006x^2 - 0,233x + 5,618$	0,773
Pine	$y = -0,032x^2 + 0,158x + 4,451$	0,765

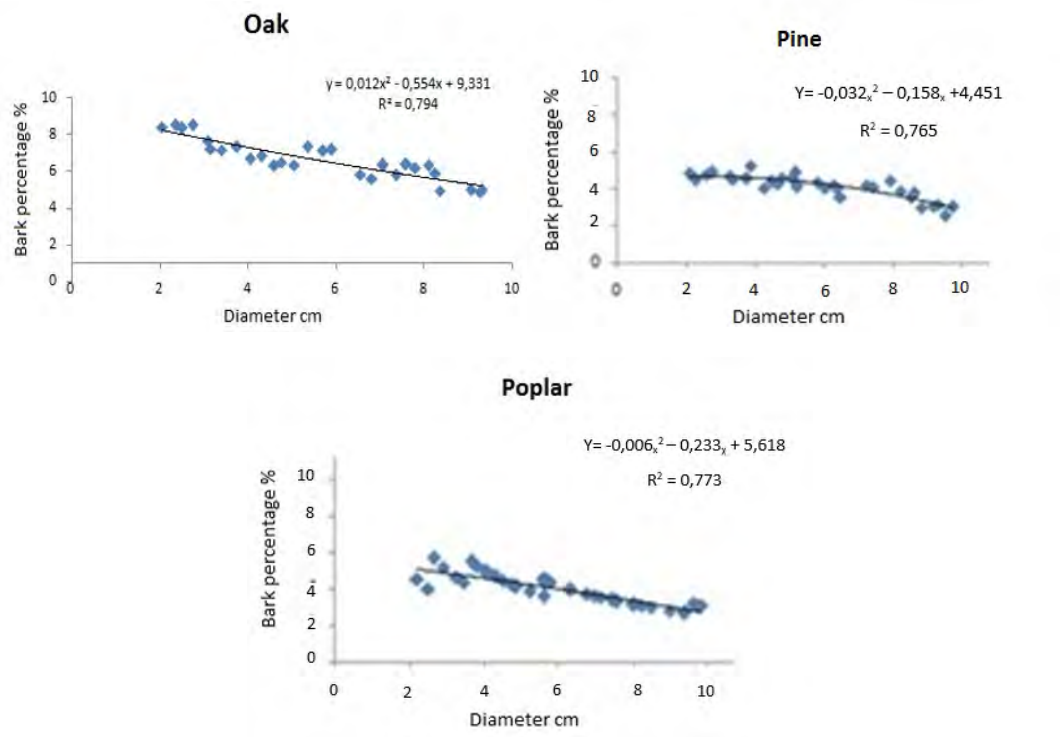

Fig. 2.
Correlation between bark percentage and branch diameter in oak, poplar and pine. Proximate analysis.

Table 3 gives ash, volatile mater (VC) and fixed carbon (FC) content of all branches, twigs and of wood and bark of thick (d=>5cm) and thin (d=2-5cm) branches of oak, poplar and pine.

Ash content

Ash content varied between the various parts of branches and between the species from 0, 38% in the wood of thick branches of pine to 8,01% in the bark of thin branches of oak. It was multiple higher in bark than in wood of branches and higher in thin than in thick branches. It is obvious that ash content increases with decreasing branch diameter (see also Table 1 and Fig. 2). Twigs in oak and pine had lower ash content than bark but higher than the all branches. The ash content of the all thick and thin branches was in oak 2,53% and 3,81%, in poplar 1,12% and 1,58% and in pine 0,79% and 1,16%, respectively. Werkelin et al. (2007) found big differences in ash content between wood and bark in branches of spruce, pine, poplar and birch. Dzurenda (2013) found 0,37% and 5,73% ash in wood and bark of poplar branches, respectively. Zeng et al. (2014) also found significant differences in ash content between different parts of Masson pine trees. Dibdiakova et al. (2015) measured ash content of different parts of scots pine tree and found that branch base has ash content of 0.48% and the branch twigs about 1.56%.The EN ISO 17225-2 (2014) standard for domestic pellets requires ash content less than 0,7% for A1 class, less than 1,2% for A2 class and less than 2% for B class pellets. Oak branches and pine twigs do not meet the above standard requirements and they should not be used alone for pellet production.

Table 3

Proximate analysis of logging residues

Property	Thick branch			Thin branch			Twigs	p-value[1]
	All*	Wood	Bark	All*	Wood	Bark		
Oak								
Ash (%)	2,53 ±0,045	1,2 ±0,035	6,96 ±0,065	3,81 ±0,645	1,33 ±0,425	8,01 ±0,495	4,14 ±0,690	+
VC (%)	79,96[d] ±0,187	80,32[d] ± 0,520	75,72[a] ± 0,177	78,84 [c] ± 0,386	80,83[d] ± 0,435	75,26[a] ± 0,859	76,95[b] ± 0,501	+

F C (%)	17,51 [b] ±0,310	18,58 [d] ± 0,433	17,33 [a] ± 0,463	17,35 [b] ± 0,455	17,85 [b] ± 0,196	16,72 [a] ± 0,337	18,20 [c] ± 0,503	+
Poplar								
Ash (%)	1,12 ±0,004	0,88 ±0,022	3,57 ±0,462	1,58 ±0,042	0,89 ±0,014	4,84 ±0,575	-	+
VC (%)	81,78 [c] ±0,570	81,37 [c] ± 0,342	79,52 [b] ± 0,404	80,89 [c] ± 0,412	81,88 [d] ± 0,345	78,64 [a] ± 0,5046	-	+
F C (%)	17,10 [b] ±0,391	17,75 [c] ± 0,255	16,95 [b] ± 0,279	17,43 [b] ± 0,156	17,43 [b] ± 0,243	16,52 [a] ± 0,326	-	+
Pine								
Ash (%)	%0,79 ±0,01	0.38 ±0,03	2,68 ±0,04	1,16 ±0,04	0,42 ±0,02	3,06 ±0,020	2,27 ±0,06	+
VC (%)	77,42 [c] ±0,332	81,24 [e] ± 0,120	73,23 [a] ±0,455	77,92 [d] ± 0,630	80,2 [e] ± 0,105	72,80 [b] ± 0,390	76,79 [c] ± 0,191	+
F C (%)	21,78 [d] ±0,280	18,38 [a] ± 0,399	24,09 [e] ± 0,275	20,92 [c] ± 0,387	19,53 [b] ± 0,236	24,15 [e] ± 0,391	20,92 [c] ± 0,211	+

*All branch (wood and bark at average diameter from Table 1) [1]Oneway NOVA variance test (p=0,05%). In each column, figures followed by different letters ([S]) indicate significant difference by Duncan's multiple range test (P<0.05).

Volatile and Fixed Carbon content

Volatile matter (VC) in oak ranged between 75,26% in the bark of thick branches and 80,83% in the wood of thin branches, in poplar between 78,64% in the bark of thin branches and 81,88% in wood of thick branches and in pine between 72,80% in the bark of thin branches and 81,24% in the wood of thick branches. VC of wood and bark was higher in poplar in all tree parts. In all species, VC of wood was higher than in bark. Fixed carbon (FC) ranged in oak between 16,72% in the bark of thin branches and 18,58% in wood of thick branches, in poplar between 16,52% in the bark of thin branches and 17,75% in the wood of thick branches and in pine between 18,38% in the wood of thick branches and 24,15% in bark of thin branches. FC in all parts of pine was higher in bark than in wood while in oak and poplar it was higher in wood. In a study (Telmo et al. 2010) of proximate analysis of 13 wood species, VC varied among the species between 74,7% and 87,1% and FC between 12,4% and 22,5%. In the same study VC and FC of oak wood was 81,7% and 18,0%, of pine 85,8% and 14,1% and of poplar 87,1% and 12,4%, respectively.

Ultimate analysis

Table 4 gives the carbon, hydrogen, oxygen and nitrogen content of the all branches, twigs and of wood and bark of thick (d=>5cm) and thin (d=2-5cm) branches of oak, poplar and pine.

Table 4

Ultimate analysis of logging residues

Sample	Thick branches			Thin branches			Twigs	p-value[1]
	All	wood	bark	All	wood	bark		
Oak								
C(%)	46,23[b] ± 0,519	46,92[b] ± ,285	45,12[a] ±0,254	46,90[b] ± 0,577	46,50[b] ± 0,345	45,22[a] ± 0,143	48,85[c] ± 0, 238	+
H(%)	6,06[b] ± 0,102	6,35[c] ± ,051	6,34[c] ± 0,051	6,09[b] ± 0,015	6,27[c] ± 0,090	5,78[b] ± 0,119	6,22[b] ± 0,040	+
O(%)	47,71	46,7	48,5	47	47,2	49	45	
N(%)	0,29[b] ± 0,020	0,21[a] ± ,010	0,217[a] ± 0,010	0,312[b] ± 0,040	0,216[a] ± 0,020	0,414[c] ± 0,010	1,173[d] ± 0,060	+
Poplar								
C(%)	45,015 [d] ± 0,095	44,02 [b] ± ,111	43,67 [a] ± 0,202	45,13 [d] ± 0,230	45,29 [d] ± 0,147	44,82 [c] ± 0,286	-	+
H(%)	6,15 [d] ± 0,121	6,16 [d] ±0,150	5,46 [a] ± 0,089	5,96 [c] ± 0,075	6,11 [d] ± 0,065	5,75 [b] ± 0,081	-	+
O(%)	48,8	49,8	50,9	48,9	48,6	49,4	-	
N(%)	0,105 [a] ± 0,005	0,195 [c] ± ,004	0,215 [d] ± 0,008	0,207 [c] ± 0,007	0,12 [b] ± 0,005	0,403 [e] ± 0,004	-	+
Pine								

C (%)	50,62 [c] ± 0,325	49,97 [b] ±0,117	50,73 [c] ± 0,340	49,94 [b] ± 0,272	49,02 [a] ± 0,125	49,75 [b] ± 0,310	50,00 [b] ± 0,48	+
H (%)	6,29 [b] ± 0,075	6,85 [c] ± ,020	6,10 [a] ± 0,045	6,54 [b] ± 0,144	6,64 [c] ± 0,123	6,36 [b] ± 0,110	6,58 [d] ± 0,04	+
O (%)	43,1	43,2	43,2	43,5	44,3	43,89	43,41	
N (%)	0,13 [b] ± 0,011	0,07 [a] ±0,005	0,595 [d] ± 0,042	0,2 [c] ± 0,020	0,13 [b] ± 0,026	0,41 [d] ± 0,020	0,76 [e] ±0,02	+

*All branch (wood and bark at average diameter from Table 1), [1]Oneway NOVA variance test (p=0,05%). In each column, figures followed by different letters ([S]) indicate significant difference by Duncan's multiple range test (P<0.05).

Carbon content varied in oak between 45,12% in the bark of thick branches and 48,85% in twigs; in poplar between 43,67% in the bark of thick branches and 45,29% in the wood of thin branches and in pine between 49,02% in the wood of thin branches and 50,62% in bark of thick branches. There were no difference between thick and thin in all branches in oak and poplar, while in pine thick branches had higher carbon content. Branch wood had higher C % than bark in oak and poplar and lower in pine.

Hydrogen content varied in oak between 5,78% in twigs and 6,35% in the wood of thick branches; in poplar between 5,46% in the bark of thick branches and 6,16%, in the wood of thick branches and in pine between 6,10% in the bark of thick branches and 6,85%, in wood of thick branches. There no difference between thick and thin whole branches in oak and pine, while in poplar thick branches had higher hydrogen content. Hydrogen content was higher in Pine than oak and poplar branches and higher in oak than in poplar branches. Ragland and Aerts (1991) noticed that the C content of softwood species varies between 50 and 53%, and that of hardwood species between 47 and 50% mainly due to the varying lignin and extractives content. They also give 52,25% and 54.9% C in oak and pine bark, respectively. Nurmi (1993) gives for trembling aspen >5mm branch wood 46,84% C and 5,96% H, and for branch bark 48,05% C and 5,77% H. He also gives for scots pine branch wood 53,53% C and 6,03% H and 54,99% C and for branch bark 54,99% C and 6,7% H. Wilen et. al. (1996) give for scots pine logging residues, 51,3% C and 6,1% H.

Oxygen content was determined by subtracting C, H, N and ash content from the whole mass (100%). In oak O% varied from 45% in twigs to 48,5% in the bark of thick branches. In poplar oxygen content varied from 48,6% in the wood of thin branches to 50,9% in the bark of thick branches and in pine it varies from 43,1% in thick branches to 44,3% in the wood of thin branches. Oxygen content was higher in oak and poplar than in pine. Oxygen content was lower than carbon content in all pine biomass components, while in oak and poplar it was higher

Nitrogen content was higher in oak and it varied from 0,216% in wood of thin branches to 1,173% in twigs. In poplar and pine N content varied from 0,105% in thick branches and 0,403% in bark of thin branches to 0,07 in wood of thick branches and 0,76 in twigs, respectively. In all cases, bark had 2-3 times higher nitrogen content than wood. Dzurenda (2013) give 0,36%, 0,65% and 0,46% N content for populous branch wood, branch bark and branch chip. Alakangas (2005) give 0,3% N content for scots pine whole trees and 0,4% for pine logging residues. Oak and pine twigs have higher N content than EN ISO 17225-2 (2014) standard for domestic pellets requires and should not be used alone for pellet production.

Heating value

Table 5 shows the heating value of the various branch components of oak, poplar and pine Heating value is given in two types, as higher heating value (HHV) and as higher heating value of ash free material (HHVf). The later was calculated after subtraction of ash from the weight of the HHV determination biomass samples. The higher heating value (HHV) of oak ranged from 18,72MJ/Kg in bark of thick branches to 19,52MJ/kg in the wood of thin branches. In poplar the higher heating value (HHV) ranged from 18,02MJ/kg in the bark of thick branches to 18,28MJ/kg in the wood of thin branches and in pine ranged from 20,75MJ/kg in wood of thin branches to 21,0MJ/kg in the bark of thick branches. In oak HHV was higher in wood than in bark, but in pine it was higher in bark. The ash free higher heating value (HHVf) ranged in oak from 19,38MJ/kg in wood of thick branches to 20,80MJ/kg in bark of thin branches, in poplar from 18,43MJ/kg in the wood of thick branches to 19,19MJ/kg in the bark of thin branches and in pine from 20,84MJ/kg in wood of thin branches to 21,60MJ/kg in bark of thin branches. HHVf increased proportionally with the removal of ash and was higher in bark than in wood in all species.

Table 5

Heating values (MJ/kg) of logging residues

Property	Thick branch			Thin branch			Twigs	p-value[1]
	All*	Wood	Bark	All*	Wood	Bark		
Oak								
HHV[2]	19.26[d] ±0.050	19.15[c] ±0.110	18.72[a] ±0.090	19.31[b] ±0.060	19.52[d] ±0.085	19.13[a] ±0.090	19.3[b] ±0.122	+
HHVf[3]	19,76	19,38	20,12	20,07	19,78	20,80	20,13	
Poplar								
HHV	18.26[c] ±0.075	18.27[c] ± 0.080	18.02[c] ± 0.010	18.27[b] ± 0.020	18.28[b] ± 0.025	18.26[a] ± 0.035	-	+
HHVf	18,47	18,43	18,69	18,68	18,44	19,19	-	
Pine								
HHV	20.95[c] ±0.020	20.84[d] ± 0.015	21.00[d] ± 0.020	20.80[a] ±0.045	20.75[b] ± 0.025	20.84[a] ± 0.025	20.95[e] ±0.015	+
HHVf	21,20	20,92	21,58	20,94	20,84	21,60	21,44	

**All branch (wood and bark at average diameter from Table 1), [2]HHV= higher heat value, [3]HHVf= higher heating value free ash, [1]Oneway NOVA variance test (p=0,05%). In each column, figures followed by different letters ([S]) indicate significant difference by Duncan's multiple range test (P<0.05).*

On the average, heating value was higher in pine biomass than in oak and poplar and higher in oak than in poplar. Philippou (1982) found for the same tree species in oak 19,65MJ/kg and 18,79MJ/kg, in poplar 19,78MJ/kg and 19,62MJ/kg and in pine 20,35MJ/kg and 21,60MJ/kg for stem wood and stem bark, respectively. Agar (2014) making pellets from pine and logging residues found that the average caloric content for the whole pine tree was 20,800MJ/kg and for the residues 21,600MJ/kg. Griu and Lunguleasa (2015) found 19,13MJ/kg for poplar stemwood.

CONLUSIONS

This study has shown that oak, poplar and pine residues left in the forest after harvesting differ in some properties that are important for energy use. Bark and ash content increased with decreasing diameter of branches. Ash content was higher in bark than in wood of branches in all species. Ash content of all thick and thin branches was in oak 2,53% and 3,81%, in poplar 1,12% and 1,58% and in pine 0,79% and 1,16%, respectively. Ash content of twigs was in oak 4,14% and in pine 2,27%. Nitrogen content of branches varied from 0,105% to 0,312% and it was higher in oak and in thin branches. N content of twigs was 1,173% in oak and 0,76% in pine. Oak branches and oak and pine twigs had ash and nitrogen content higher than that required by the EN ISO 17225-2 (2014) standard (Howard 1988) for domestic pellets and they should not be used for energy, at least for pellets production. Volatile mater, fixed carbon, carbon and hydrogen content were in the range given by other researchers. Heating value ranged between 18,27MJ/kg to 21,0% and it was higher in pine than in oak and poplar, and higher in twigs and thin branches.

From the above results we could conclude that branches of poplar and pine could be good material for domestic pellet production and other energy usages. Branches of oak and twigs should be left to provide nitrogen and minerals to the forest soil. Utilization of logging residues biomass could create business opportunities and employments in local populations, generate profit from residual material and provide energy self-sufficiency for rural communities.

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RESEARCH ON BRIQUETTES OBTAINED FROM SHREDDED TOBACCO CIGARETTES, AS A LIGNO-CELLULOSE FUEL

Aurel LUNGULEASA, Cosmin SPIRCHEZ and Adriana FOTIN
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: lunga@unitbv.ro
E-mail: cosminspirchez@unitbv.ro
E-mail: adrianafotin@unitbv.ro

Abstract

The paper presents some considerations about briquettes derived from waste shredded tobacco cigarettes, used as a ligno-celluloses fuel. A synthesis of main advantages and disadvantages of tobacco plants is presented. Physical and calorific values of these types of briquettes are presented according to their different methodologies. If density and calorific value fall into the European standardized restrictions, ash content exceeds these restrictions due to the torrefaction process of tobacco manufacturing and the fact that tobacco actually is an agricultural plant (cereal and other agricultural plants usually have an higher ash content). Finally it is concluded that the briquettes obtained from shredded tobacco can be used moderately as fuel for stoves and furnaces.

Key words*: tobacco; briquette; ash content; calorific value.*

INTRODUCTION

Tobacco cigarette consumption has increased worldwide in recent years, despite negative advertising imposed by the European community and other international organizations. In particular the illegal sale of cigarettes has increased, mainly due to soaring prices caused by the imposition of customs duty and tax. As a result there are increasing quantities of cigarettes seized by authorities that must be destroyed. Destruction of these cigarettes is necessary because there is no company stamp and thus no legal liability imposed. As a result all consumers can have their lives threatened. Usually the seized cigarettes must be destroyed by shredding, following disposal in landfill by companies who are specialized in this field. These companies can recycle the remnants of tobacco, filters and paper resulting from shredding and can, for example, produce briquettes to be used in combustion. To be able to use them in good condition in heating boilers and stoves, cigarette briquettes made from chopped material had to be analysed for physical, mechanical and calorific value. Also, before knowing the characteristics of briquettes it is necessary to know the components of cigarettes, especially tobacco leaves, with form the majority by over 80% by mass.

Tobacco has the botanical name *Nicotiana species* L, Solanaceae family. Worldwide there are about 70 species of tobacco, from which *Nicotiana tabacum* is the most common, followed by the stronger one *Nicotiana rustica*, both of which originate from the USA. Tobacco can grow on the same land for over 15 years, without apparent fatigue of the soil (TTG 2017). Due to its sensitivity to cold, seeds are first germinated on a warm seedbed (with fresh manure), then planted in the field. Before planting, seedlings are prepared by reducing total exposure and contact with the outside air both during the day and the night. Planting is done in cooler periods of the day, morning and evening in April and May (for conditions of Romania and other countries of South-eastern Europe). Weed control is realized with herbicide and/or by performing manual or mechanized weeding. Before harvesting, the cutting of the flowers is recommended, to avoid the leaves growing too vigorously (Fig. 1).

Leaf tobacco harvesting is done manually in several episodes, by peeling off the 5-6 leaves from the central stem. After harvesting, the leaves are kept for 2 hours in a pile for a slight wilting, so that leaves do not break during subsequent manipulations. Leaf tobacco drying is done in several stages, the first stage being their yellowing, which is obtained under conditions of constant temperature and humidity, in order to reduce the moisture content of the leaves from 80% to 40%. Drying can be made natural or artificial, in a shaded area and without air flows. Tobacco is placed in bundles of 10-15 sheets of leaves (Fig 2). Current production is 1000-2000 kg/ha. In the production process of cigarettes the torrefaction operation of leaves is used, employing a thermal process at high temperatures above 200^0C, in order to intensify the flavour of the tobacco.

Fig. 1.
Plant of tobacco and its inflorescence.

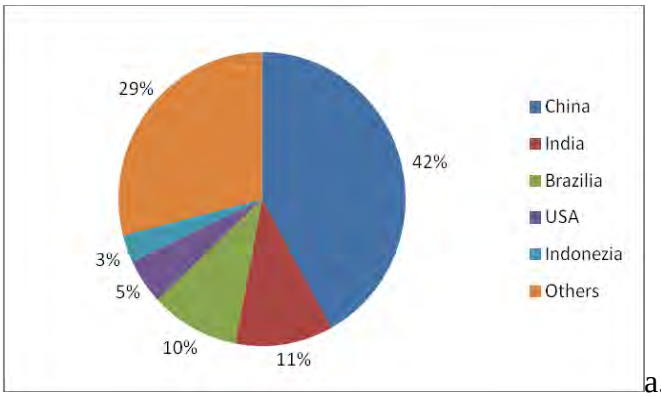
Fig. 2.
Leaves of tobacco in time of naturally drying.

Tobacco dried leaves are smoked as cigarettes, cigars and in pipes. Using cigarettes generally has both advantages and disadvantages. The main disadvantage is considered that smokers have a risk factor for many diseases that are potentially life-threatening, such as those affecting the heart, liver and lungs, especially the onset of cancers. The World Health Organization sets forth that smoking is the biggest cause of premature death in the world (Stefani 2010). Figure 3 shows the top of 5 global manufacturers of tobacco. It notes that big countries are in the top position, like China, India, Brazil and the USA. Tobacco cigarette consumption is however different when referring to the number of inhabitants of the country of reference, the first places being taken by the Czech Republic and South Korea, followed by Germany, Switzerland and Turkey (as at 2011).

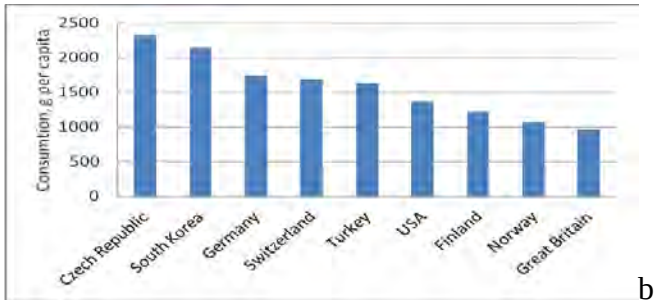
Tobacco has long been used as a medicinal plant in South and North America, being consumed in many forms such as chewing, smoking, snuff, eating, drinking, and body anointments and in the form of eye drops (Stefani 2010). Tobacco popularity is due to the fact that small amounts of tobacco produces a mild stimulating effect on the functions of the user, while large quantities can produce hallucinations and even deep trances. Tobacco was also a guide to health and healing, had mystical connections and was part of ancient cultures and civilizations. Some American tribes have used tobacco to cure ailments such as ear pain, snake bites, cuts and burns, respiratory disease, fever, seizures, nerve disorders, urinary and skin diseases. In Europe, tobacco was introduced after its discovery in America by Christopher Columbus in 1492, by Jean Nicot in 1585, the French ambassador in Portugal, after whom the name nicotine was adopted (Vlaescu 2011). At first the tobacco was used as a decorative element (due to special and beautiful blossom), then for its medicinal properties. The main compound of tobacco is nicotine which is an alkaloid that has a relaxing effect and stimulates the person consuming it by increasing levels of dopamine and serotonin.

Nicotine is an anti-inflammatory and has been scientifically proved that it can treat Alzheimer's and Parkinson's disease or may cause their delay (TTG 2017). When ancient tribes make peace, they smoke the peace pipe.

Over time briquettes were made from ligno-cellulosic stems of tobacco (Xinfeng et al. 2015, Peševski et al. 2010) due to their properties but never from destroyed cigarettes because there was insufficient quantity.



a.



b.

Fig. 3.
Top 5 big world-wide producers of tobacco on 2012 (a) and annual consumption per capita (b) (adaptation from Wiki 2017).

OBJECTIVE

The main objective of the present research is to investigate the possibility of using briquettes made of tobacco cigarettes, in order to use them as ligno-cellulose fuel in stoves and furnaces.

MATERIALS AND METHOD

Some briquettes were made from shredded remains of tobacco cigarettes, experimentally on a hydraulically driven machine. These briquettes were graded related to EN ISO 17225-2: 2014. The density of cigarette briquettes was determined as the ratio between the mass and volume of briquettes in each piece. Taking into account that the briquettes form is cylindrical, it follows that their density is determined by the following relationship:

$$\rho = \frac{4 \cdot m}{\pi \cdot d^2 \cdot l} \left[\frac{kg}{m^3}\right] \quad (1)$$

where: m is mass of briquettes, in g; d-diameter of briquettes, in mm; l-length of briquettes, in mm.

Prior to determining the length, the briquettes were partitioned to 5mm length and the ends were polished in order to measure length with high precision, by using a calliper with an accuracy of 0.01mm. A minimum of 10 samples were taken from five different long briquettes.

From the each briquette sub-samples were taken by cutting pieces of 0.6-0.8g in order to determine calorific values, using a XRY-1C bomb calorimeter, (made in China.) The calorific value is expressed as the amount of heat released from burning of mass unit. The relationship for determining the calorific value is as follows:

$$CV = k\frac{(t_f - t_i) - q}{m} \quad [MJ/kg] \quad (2)$$

where: there are: k- calorific characteristics of installation, determined by calibration with benzoic acid; t_f - final temperature, in ^{0}C; t_i - initial temperature, in ^{0}C; m -mass of briquette sample, in g; q– heat obtained during the combustion of the nickel-chromium alloy and cotton wire.

The calorific characteristic of the installation is determined by calibration, using benzoic acid encapsulated every of 0.5 or 1g, having a known calorific value (usually 26.463 MJ/kg). In fact in relation 2 it is known to have a calorific value *CV* of acid, the heat given off by wire of nickel – chromium alloy and cotton noted with *q*, and mass of nitric acid *m*. During the calibration test the two temperatures t_f and t_i, are determined the only unknown value that the device will determine being its characteristic *k*. This characteristic will be used for the next set of about 20-50 tests, but for a period not exceeding 30 days.

For the determination of ash content, the material is prepared by chopping and sorting shredded material with a 1 × 1mm sieve. The test material taken is that fraction that passes through the sieve. Some portions that are dried to constant mass in a laboratory oven are taken from the sorted material. For all tests a crucible made of an alloy resistant steel is used, which is prepared for testing by weighing and repeated drying up to 4-5 times until constant weight is achieved. It was weighed with 3 decimal place precision. All material pieces were retained within the crucible so as to not lose material after burning. Also, to protect the laboratory oven that works at 800 ^{0}C, all smoke is eliminated by primary burning over a gas flame. The test is considered terminated when sparks are no longer seen in the ash and that is a light grey, but certainly for not less than 20 minutes. Usually, ash content is determined as the ratio between the amount of ash (m_a) and the initial mass sample of the resulting ash (m_{s0}), i.e.:

$$A_c = \frac{m_a}{m_{s0}} \cdot 100\ [\%] \tag{3}$$

Relationship (3) is valid only for a moisture content of 0% for both sample (powdered raw material and the resulting ash). If working with certain initial moisture of initial dust, especially when there are more tests, it is necessary to make changes in the relationship (3). It takes into account the overall relationship of absolute moisture content MC, determined by the ratio of water content mass and mass of oven-dry material:

$$MC = \frac{m_m - m_0}{m_0} \cdot 100\ [\%]$$

The necessary calculations to extract m_0 are made:

$$MC \cdot m_0 = 100 \cdot m_m - 100 \cdot m_0$$
$$MC \cdot m_0 + 100 \cdot m_0 = 100 \cdot m_m$$
$$m_0(MC + 100) = 100 \cdot m_m$$

From the above last relation, the m_0 value is extracted:

$$m_0 = m_m \cdot \frac{100}{100 + MC}$$

By introducing this value in the above general relationship of ash content (3), obtain the next relation can be obtained:

$$A_c = \frac{m_a \cdot (MC + 100)}{m_m}\ [\%] \tag{4}$$

where: m_a-mass of ash, in g; MC-moisture content, in %; m_m-mass of moist sample, in g.

Knowing the mass of the crucible m_c, the ash content of the tobacco briquettes could be determined by the following relationship:

$$A_c = \frac{m_s - m_c}{m_a - m_c} \cdot 100 \quad [\%] \tag{5}$$

where:

m_{si} - the mass of the initial dried sample with the crucible, in g;

m_c - the mass of the empty crucible, in g;

m_{sf} - the mass of the final dried ash with the crucible, in g.

The ash content must be determined for at least 10 valid samples. Any sample or tests that were susceptible to errors had to be eliminated.

RESULTS AND DISCUSSION

Since the briquettes made from cigarettes contain paper, filter (cellulose acetate) and graded tobacco, all determinations were performed separately for each component and for the briquettes containing all three main components in the mixture. The main element is tobacco, the other two

components having only a weight percentage of 15-20%. Moisture content of all samples taken from briquettes stored in polyethylene film was determined referring to EN ISO/IEC 322, by drying and weighing method. The value of moisture content was around 10%. In making relevant comparisons the calorific and ash content values were extracted for some combustible products, which are presented in Table 1.

Table 1

Comparative values of calorific value and ash content

Combustible material	Calorific value		Ash content
	MJ/kg	Kcal/kg	
Fire wood, beech and hornbeam	18.400	4397	0,2-1.2%
Vegetable straws	16.000	3824	3-8-12 %
Pellets and briquettes	18.900	4517	0,3-0,8%
Superior fossil coals	29.400	7026	2-3%
Petrol	46.200	11042	0.8-1.3%
Methane gas	35.170	8405	------

Briquettes density was determined based on the European standard EN ISO/IEC 323: 2005 and the averaged value of 914 kg/m^3 was obtained, above the reference standard ÖNORM M7135 product of 860 kg/m^3. Density of filters and paper briquettes was lower, just under 900 kg/m^3.

The calorific value of the components and the mixture is shown in Table 1. It is observed that the calorific value of paper and filters is slightly higher, but the lowest calorific value of purely tobacco is due to the elimination of exothermic volatiles during the torrefaction process of tobacco. Overall the calorific value limit imposed is within the standard product over 17.5 MJ/kg.

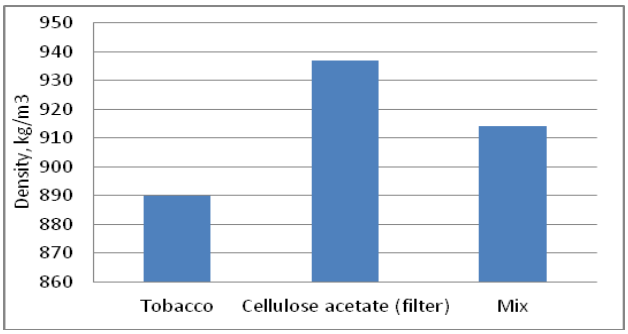


Fig. 4.
Density of briquettes.

Ash content of briquettes was very high in pure tobacco, small for a mixture of paper and filter and large for the mixture of the three components. In general, ash content exceeded the upper limit prescribed by the product standard, up to 6%. This is due to two main causes. Firstly, tobacco is a plant that has a higher ash content that wood. Secondly, in the manufacturing process, the torrefaction operation of tobacco occurs, which significantly increases its ash content, by removing volatiles.

Table 2

Calorific value and ash content of tobacco briquettes

Features		Mixture	Filters and paper	Pure tobacco
Calorific value, MJ/kg	super	17.652	17.654	13.051
	infer	17.210	17.213	12.632
Ash content, %		15.0	6.0	19.2

Because these briquettes have high ash content and this adversely affects their calorific value it is necessary to analyse the influences using the following relationships:

$$CV_{Mc} = CV_0(1 - Mc - 0.1 \cdot A_c) \quad [MJ/kg] \tag{6}$$

where: CV_{Mc}- calorific briquettes at certain moisture content, in MJ/kg; CV_0- the calorific value of the dried material, in MJ/kg; Mc-moisture content, in decimal units; A_c-ash content, in decimal units.

High ash content exists in all lignocelluloses plants, but also in tree bark. Given the calorific values obtained from testing of mixture samples (17.652 MJ/kg), an average moisture content of

briquettes 10% and using the relation (4), a mathematical model can be made using Excel program, that shows the influence of ash content on calorific mix briquettes, as shown in Fig. 5.

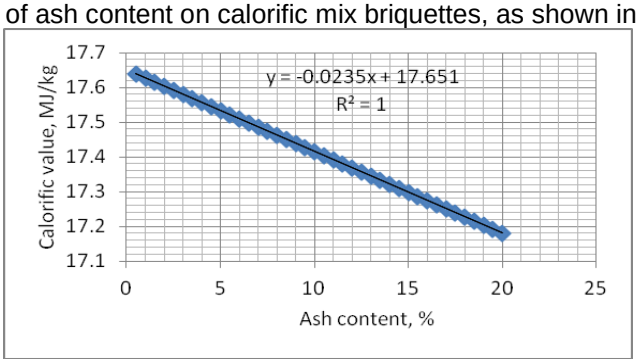


Fig. 5.
Influence of ash content on calorific value of tobacco briquettes.

It can be seen that there is a linear influence of ash on the calorific content, from a calorific value of 17.65 to 17.18 MJ/kg, i.e. a decrease by 2.6% of calorific value due to the 20% ash content. This is explained by the fact that the minerals have a great amount inside of lignocelluloses plants. Large quantity of ash and minerals reduces lignin content (chemical compound that is responsible for calorific value increasing). Similar considerations were found by other authors on some cereal plants (Xinfeng et al. 2015, Peševski et al. 2010) or wood-bark sawdust (Sotannde et al. 2010, Hytonen and Nurmi 2014).

CONCLUSIONS

Briquettes made from the remains of disintegrated tobacco cigarettes can be used as fuel due to their combustible properties even if these are modest. If the low price of these briquettes compensates for their low calorific performance, then briquetting of cigarette waste is a step forward compared to sending them to landfill, when they will be decomposed by nature. It is recommended to use these briquettes with caution because of noxious gaseous emissions produced by burning and to use tobacco mixed with sawdust (for increasing calorific power, compactness and reduced ash content), the latter procedure remaining the only way to boost the performance of such briquettes (and constitutes new direction of research).

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SECTION 11. ECONOMICS IN WOOD INDUSTRY

COMPETITIVE STRATEGIES TO WOOD PRODUCTION IN EUROPE – A CONCEPTIONAL STUDY

Johann TRISCHLER
Luleå University of Technology, Wood Science and Engineering, SE-931 87 Skellefteå, Sweden
E-mail: johann.trischler@ltu.se

Dick SANDBERG
Luleå University of Technology, Wood Science and Engineering, SE-931 87 Skellefteå, Sweden
E-mail: dick.sandberg@ltu.se

Abstract

Wood is a raw material with multifaceted properties making it useful for a diversity of industrial products. However, its primary production is restricted by environmental conditions and alternative land use. Forestry, as the main supplier of wood as raw material, is assumed to act strategically with regard to the market situation and to try, therefore, to provide the most beneficial mix of different assortments of wood. Changes in the supply of different assortments and their current value on the market are assumed to be closely related. Fluctuations in stock and market value affect the wood-using industry depending on the different assortments as raw material.

The purpose of this study was to apply a concept of competitive strategies for market-driven goods to forest management in Europe, in order to derive information about the operational possibilities of forest management in specific market situations. For this purpose, Steinmann's and Schreyögg's concept of competitive strategies for market-driven goods was applied to forest management, in order to defining different competitive strategies for the production of different assortments of wood. The results show that different market strategies can be applied to forest management and that, even though the overall production is limited, forestry has quite a high flexibility regarding the competitive strategy used. The results also show possible impacts on the nature of forests due to changes in forest management.

Key words: *assortment of wood; competitive strategy; forest management; forest production; market condition.*

INTRODUCTION

Forests provide a multitude of products and services which have an ecological, socio-cultural or economic value (de Groot et al. 2002; Schmithüsen 2007). The mix of services available depends on biological characteristics and on the nature of the economic regime. The use of some goods is market-driven, others are used under a variety of agreements, and others are available to communities as free goods (Chopra and Kumar 2004; Schwarzbauer et al. 2015). Wood is one of those goods for which production is market-driven, see e.g. Vaux and Zivnuska (1952). Its supply includes different assortments suitable for diverse purposes in the wood-using industry, depending on different forest management strategies (Burschel and Huss 1987; Wagenführ 2007). The term "assortment" stands for a selection of roundwood characterized by a specific length, diameter and wood properties, such as saw logs or veneer logs, pulpwood, wood for energy conversion including small diameter trees or parts of trees such as tops or branches, of either softwood or hardwood.

Strategic planning is necessary for any business organisation and, for forestry in particular, strategic long-range planning goes back to the 18th and 19th centuries when concern about a continuous future wood supply in Central Europe came to the fore (Hoogstra and Schanz 2008). Historically, the different ways of using the forest in combination with the dynamics of the environmental parameters led to continuous changes in the nature of the forests (Farrell et al. 2000; Gamborg and Larsen 2003; McGrath et al. 2015). The complexity of forest management makes it difficult to investigate strategies for land availability, timber sustainability and harvest strategies, and this means that, for forest models for strategic management, a trade-off between their complexity and their usability for strategic planning must be found. Mostly, it has to be assumed that forest production does not change with forest management. Models used today also tend to ignore issues of prices and markets for forest products (Gunn 2007).

According to Porter (1980), the survival of an organisation in a competing environment lies in the creation of the best fit between environment and resources, which expresses itself in three core generic competitive strategies: cost leadership, differentiation and focus. Cost leadership is the cutting

of costs to levels lower than those of competitors which gives an above-average cash-flow even with relative low prices for the products. Differentiation is achieved by innovation or customisation to its location and leads to lower user costs or an increase in the use value of a product. Limitation on key aspects means that a company focuses on different market niches or core markets regarding customers or regionalism (Schroeter 2013). Steinmann and Schreyögg (2005) extended Porter's theory adding a third dimension to the place and focus of competition, viz.: the rule of competition. Therefore, their concept is more complex and in general illustrated graphically as a so-called strategic cube. To apply such a tool of strategic management to forestry has not yet been attempted, due perhaps to the complexity of forest management or to the fact that forests in general provide a mix of diverse goods.

OBJECTIVE

The purpose of this study was to apply "Steinmann's and Schreyögg's concept of competitive strategies" for market-driven goods to the common forest management practices in Europe, as a tool to evaluate changes in the assortments of wood supplied and the nature of forests in connection with changes in forest management strategies.

METHOD

Main parameters influencing the production and properties of wood

1) The biometric aspect of wood production

Under given climate conditions, some species show faster growth rates than others, especially in younger age classes, and this favours their use for biomass production (Burschel and Huss 1987). Species with faster growth rates in younger age classes are preferred because of the relationship between the total growth of a species, its current annual increment (CAI) and its mean annual increment (MAI). If the focus is on biomass production, a stand should be harvested when the MAI reaches its maximum. At this point, the growth curve has its second inflexion point and the CAI and MAI curves intersect each other. After that point, the increment decreases and the growth curve flattens out and approaches its maximum (Bachmann 2008). The gradient of the growth curve is steeper for fast growing species in young age classes than for species which reach their maximum later. This leads to earlier culmination points for the CAI and MAI and therefore shorter rotation cycles. This explains the benefit of such species when the focus is only on biomass production volumes regardless of the physical properties of the raw material.

On the stand level, the increment of a species is related not to the single tree but to a defined area, which means that the increment is in general that of a community of trees. To achieve high yields in biomass production, there must be a trade-off between the increment of the single tree and the stand density or number of trees, as single trees with larger crowns show a larger increment than smaller trees, whereas the stand growth of larger trees generally decreases with increasing age of the trees (Utschig 2002). Pretzsch (2006) has shown that the growing area efficiency increases with increasing stand density but that the stand growth decreases because of the large number of small, inefficient trees. If the number of trees is relatively small, the growing area efficiency is low but the stand growth is high because of the larger number of average-sized trees which have a higher efficiency. This means that dense stands have a high growing area efficiency, whereas sparsely stocked stands a have high stand growth. Not only the position of the single tree within the stand but the mixture of species also influences the production per area. It has been shown, based on modelled data, that in the temperate zone the increment of a stand can vary between -30% and +40% depending on the mixture of species (Pretzsch 2012).

2) Site factors influencing the wood properties

Water has a strong impact on the growth rate in temperate regions, especially in the form of precipitation. If water is not the limiting factor for growth, nutrients are becoming the limiting factor. The effects of water and nutrients as limiting factors have been shown for species like Norway spruce grown in Sweden (Bergh et al. 1999) and Maritime pine grown in south-western France (Trichet et al. 2008).

Temperature is also important for the growth rate and is one reason why the ratio of broad-leafed trees to conifers in the sub-boreal and temporal climate zones differs from that in the tropical climate zone. The larger proportion of conifers in the cooler zones suggests that conifers have a faster growth rate under cooler conditions (Way and Oren 2010). The strong effect of temperature in combination with precipitation on the growth of a tree has been shown in stands of Norway spruce (Larson 1969; Šoškić et al. 2003). Taking into consideration the results reported by Nylinder and Hägglund (1954) and by Bergh et al. (1999), the conclusion is that temperature has not only a direct but also an indirect effect on the growth rate. A low temperature leads to a slower mineralisation

process, and the lack of nutrients can have a negative impact on wood properties, as the Swedish expression "starvation wood" testifies (Nylinder and Hägglund 1954). It has been found that with increasing latitude not only the width of the annual rings but also the amount of latewood and the mean fibre length have decreased. It has also been shown that not only very wide but also very narrow annual rings lead to a decrease in density (Nylinder and Hägglund 1954; Larson 1969; Thörnqvist 1987) which indicates that the climate has a strong impact on wood properties. The density of softwoods is determined by the proportion of earlywood to latewood and this proportion depends to some extent on the width of the annual rings (Brazier 1970). A larger proportion of latewood with its thicker cell walls (Grahn et al. 1995) leads to a larger amount of S2-layers which amount to 70 vol.% of the fibre wall (Forsberg 1997) and the highest cellulose content (Sandberg et al. 2011).

Fertilisation and thinnings in Douglas fir, Norway spruce and Scots pine stands showed differences in wood properties between the wood of treated trees and the references (Ericson 1966; Jozsa and Brix 1989; Barbour and Kellogg 1990). Kyrkjeeide et al. (1994) showed that the smaller proportion of intermediate and mature wood in fast-grown than in slow-grown Norway spruce lead to a lower quality classification at a given diameter when sorted after sawing-drying-ripping. Wood of fast-grown Norway spruce had a lower bending strength than slowly grown Norway spruce (Eikenes and Lackner 1990). The relationships between juvenile wood, mature wood and density differ not only with the age of the tree within the tree diameter but also within the height (Ericson 1966; Larson 1969; Jozsa and Brix 1989; Jozsa and Sen 1992; Thörnqvist 1993; Kennedy 1995). In Sitka spruce stands, it has been demonstrated that the wood properties can be influenced by silvicultural operations such as spacing, pruning, fertilizing and irrigation which have an impact on the frequency of knot incidence (Brazier 1977; Brazier et al. 1985; Macdonald and Hubert 2002).

The concept of competitive strategy in the context to forestry

For an organisation, a competitive strategy regarding market-driven goods has to include: cost leadership, differentiation and limitation on key aspects (Porter 1980; Schroeter 2013). Steinmann and Schreyögg (2005) extended Porter's theory with one more dimension, leading to three dimensions which can be illustrated as a strategic cube, Fig. 1. Each form of competition is defined by two characteristic variables which allow eight different combinations of the characteristic variables and therefore eight different competitive strategies for market-driven goods.

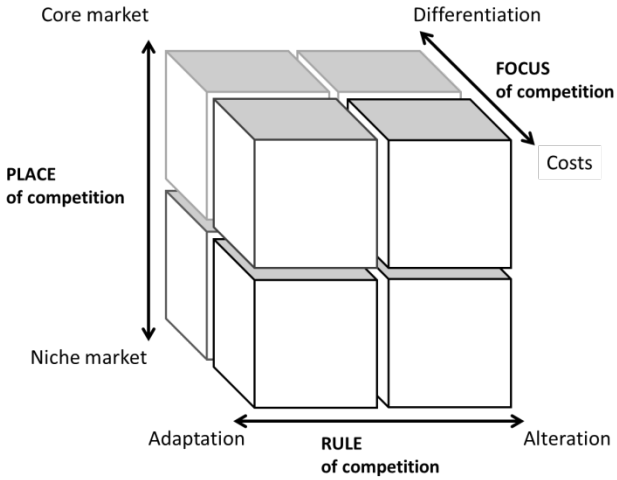

Fig. 1.
The principle of Steinmann's and Schreyögg's strategic cube after showing the three forms of competition with their two characteristic variables, the combination of which give eight different competitive strategies for market-driven goods.

A company will choose a strategy which ensures its current profit or a profit maximisation which is acceptable and realizable. Monetarily, such a selection of strategy can be interpreted as an investment, which means that the strategy with the highest capitalised value or rate-of-return should be chosen (Schroeter 2013).

In forestry, the specific site conditions in combination with diverse management strategies and intensities lead to the production of different volumes and assortments of wood (Burschel and Huss 1987). The management strategy with regard to market-driven goods such as different assortments of wood is strongly related to the market situation which reflects the current trend of use (see e.g. Mason

2007; Schwarzbauer and Stern 2010; Trømborg and Solberg 2010). This has an impact on the nature of forests (see e.g. Mason 2007; McGrath et al. 2015). Therefore, the environmental conditions can be seen as the limiting factors for the production capacities and the forest management strategies to decide which assortments (goods) out of a possible mix are produced or promoted.

Since forest management strategies have the potential to influence the output of different assortments, and since it can be assumed that wood is a market-driven good, it is possible to impute a competitive strategy to forest management. The challenge is, however, that forestry often provides a mix of different assortments. To apply the concept of Steinmann and Schreyögg, the three forms of competition – place, focus, and rule – have to be put in context with forestry. In this study, the interpretation made with regard to the current market-driven production of different assortments of wood is shown in Fig. 2 and was as follows:

(a) Place of competition
- Core market Relatively long rotation times leading to high stands, timber forests
- Niche market Relatively short rotation times, coppice and short rotation coppice

(b) Focus of competition
- Differentiation Focusing on assortments of wood with beneficial properties
- Costs Focusing on productivity, assortments of low to average wood properties (bulk goods)

(c) Rules of competition
- Adaptation Tendency to follow current market trends, specialisation
- Alteration Conservative performance, traditional forest management, variation

RESULTS AND DISCUSSION

If considering wood as market-driven goods of which the volumes and properties can be influenced by forest management strategies leading to different amounts of different assortments of wood, the concept of competitive strategies according to Steinmann and Schreyögg (2005) can also be applied to forest management. The results interpreted according to Steinmann's and Schreyögg's concept are presented in Fig. 2, showing the competitive strategies for the different assortments and different types of forest.

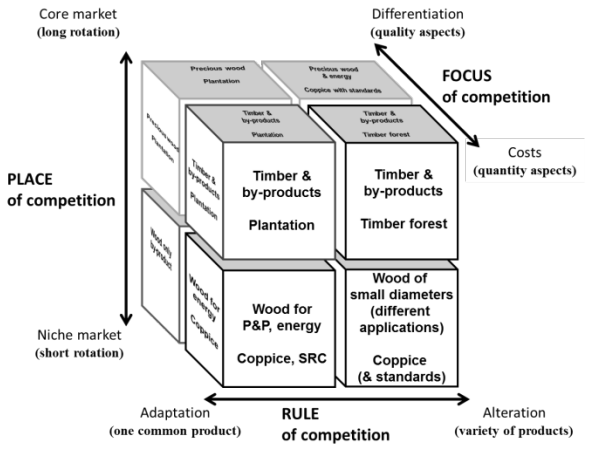

Fig. 2.
The strategic cube showing the competitive strategies for the different assortments of wood and different types of forest defined by: length of rotation time (place of competition), specialisation vs. diversification (rule of competition), and productivity vs. wood properties (focus of competition).

This study assumes that wood, like other resources, is available only in limited amounts, so that a change within the wood-using industry affects not only the competitors within the industry but also forestry which is the supplier. Abt et al. (2012) showed that an increasing demand for a particular assortment (wood for bioenergy) has an impact on the traditional wood-using industries such as saw-log processing industry and on the use of land. The magnitude of the impact depends on the shift in the levels of demand and on the supply response. The important role of forestry and agriculture as a producer of raw material is also obvious in the review presented by Berndes et al. (2003), who state that the greatest uncertainties in energy-crop production are the availability of land and the level of

biomass production. Trømborg and Solberg (2010) were able to show in a case study for Norway that an increase in the price of wood for energy purposes can affect other sectors of the wood-using industry. Similar results were reached by Schwarzbauer and Stern (2010) when simulating a rising demand for wood for energy purposes in Austria. According to their study, forestry and sawmills would profit from the greater demand and higher prices, while the wood-based panel and the pulp and paper industries would suffer.

Depending on the focus of the raw material production, silvicultural management leads to different types of forests. If the focus is on timber production, the longer rotation cycles lead to the typical "timber forests" with trees with large heights and diameters. On the other hand coppice forests with maximum rotation cycles of 10 to 30 years show completely different characteristics. A special form of coppice providing raw material for energy purposes is the short rotation coppice, which has a rotation time of even less than 10 years. Coppices with standards are a combination of these two main forest types, the timber forest and the coppice forest, with two crown layers. The upper layer is composed of trees for timber production and the lower layer of trees mainly for energy production (Burschel and Huss 1987; Bürgi 1999).

It has been assumed that forest management operates strategically regarding the production of different assortments of wood. However, regarding the production of the different assortments, it is difficult to argue that a specific forest management strategy is focusing on a single main assortment as often a mix of different assortments is provided. Nevertheless, the results also give space for more wider interpretation:

Firstly, the nature of forests and forest management is dynamic and this is important to consider when modelling (future) availability and accessibility of different assortments. It is critical to take the status quo and assume a change in available quantities without considering possible changes in forest management or in the nature of the forest. From a market-driven point of view, different trends on the market lead to changes in forest management affecting the composition of the assortments and the types of forest by a shift in the focus of production towards a new competitive strategy.

Secondly, the most critical parameters for the production of wood are the limited land area and the climate. As the production area is limited, a trade-off regarding accessibility between the different assortments has to be made. Therefore, only one single assortment can be produced as the main assortment on a given site at a given time when the production is maximized, which can be interpreted as a direct competition for the available land for production. Increasing demand requires a higher production capacity on the available land area and this leads to a shift in the direction of homogeneity. This would lead to monocultures or plantations of either soft- or hardwoods, depending on the range of assortments needed.

Thirdly, with regard to the production of different assortments, increasing market orientation leads primarily to an increase in harvest pressure (Nabuurs et al. 2006) and further to a shift towards more efficient production such as plantations, see e.g. (Bowyer 1995; Carle et al. 2002; Payn et al. 2015). Timber forests can theoretically change into coppices or coppices with standards if, for example, it becomes more profitable to be produce wood for energy purposes than wood for timber or vice versa. However, this theoretical assumption also clarifies that there is still a high potential for increasing the volume of production for different assortments, since production efficiency can be increased by concentrating on a single assortment per area. This would relativize the arguments regarding the shortage of wood and is supported by the literature in the context of forest plantations. Even if the facts are obvious, regionally forest must not change at all as global trade and intense production in climatically favourable regions can compensate high volumes of demand (Jonsson 2011; Buongiorno and Zhu 2014). However, there is still a research gap regarding econometric studies considering the multi-country market aspect (Toppinen and Kuuluvainen 2010).

CONCLUSIONS

Wood is an important raw material for many products. The future development of the supply and demand for different assortments of wood is therefore of great interest. The aim of this study was to apply "Steinmann's and Schreyögg's concept of competitive strategies" for market-driven goods on the common forest management practices in Europe with regard to the production of different assortments of wood. If the production of different assortments is market-driven, it can be assumed that forest management will follow competitive strategies in the production of different assortments. Forest management, the nature of the forest and the accessible volumes of the different assortments are changing depending on the market situation. Moreover, the results show that the production of different assortments per area is limited. Maximising the production per area is possible only for a single assortment at the expense of other assortments. Overall, the findings of this study are a useful

base and provide constructive support for diverse types of forest models with regard to the future development of wood accessibility.

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POSSIBILITIES OF IMPLEMENTING A FOREST CERTIFICATION SYSTEM FOR THE CONSERVATION AND SUSTAINABLE MANAGEMENT IN SMALL OAK STANDS FOR USE IN COOPERAGE

Ignacio J. DIAZ-MAROTO
University of Santiago de Compostela, Higher Polytechnic School
Campus Universitario s/n, 27002 Lugo, Spain
Tel: 0034 982 107531, Fax: 0034 982 823001, E-mail: ignacio.diazmaroto@usc.es

Pablo VILA-LAMEIRO
University of Santiago de Compostela, Higher Polytechnic School
Campus Universitario s/n, 27002 Lugo, Spain
E-mail: pablo.vila.lameiro@usc.es

Adriana CONDE-FERNANDEZ
University of Santiago de Compostela, Higher Polytechnic School
Campus Universitario s/n, 27002 Lugo, Spain
E-mail: adriana.conde@usc.es

Abstract

The European Union Forest Law Enforcement Governance and Trade (FLEGT) Action Plan sets out actions to prevent the import of illegal wood into the EU, to improve the supply of legal timber and to increase demand for wood coming from responsibly managed forests. The management of certified timber means prevents these practices with the environmental benefits, and with the commercial advantages for a best public image. Now, products derived from sustainable timber procurement policies are accepted, which include a legal compliance, cost savings, local innovation and development of potential markets, the creation of local green jobs, and their contribution to sustainability global. The present study was designed to evaluate the possibilities for implementing a forest certification system for small Galician oak stands with areas under 25 ha. This process must be conducted through Galician Group of Forest Certification and Chain of Custody (CFCCGA), main certification institution in Galicia, Spain. The certification system managed by the CFCCGA is the Programme for the Endorsement of Forest Certification (PEFC). The subsequent use of these stands will be the production of staves for the manufacture of barrels used for the whisky aging.

Key words: *oak wood; oak staves; cooperage industry; forest certification; Galicia.*

INTRODUCTION

Local governments and public institutions around the world realize that sustainability in procurement is a key responsibility and an important element in forward-looking policies and activities. It's an integral part of the role of the public sector to contribute to the aspirations of their constituency and to meet their needs within the limits of our planet (PEFC 2012). Aware of the importance of promoting Sustainable Forest Management (SFM), national and local governments have made sustainable timber procurement a key requirement of public purchasing (ProForest 2006). Many have put in place regulatory frameworks and legislation to this effect to tackle illegal logging and help prevent illegally harvested wood or timber from unsustainable sources entering the market. The certification systems are accepted by public procurement policies globally as providing evidence for sustainability and legality. Either system for forest certification includes the following requirements (Baharuddin and Simula 1994): i) safeguard ecologically important forest areas; ii) protect and enhance biodiversity; iii) prohibit most hazardous chemicals; iv) prohibit genetically modified trees; v) respect the rights of workers and indigenous people; vi) encourage local employment; vii) comply with basic International Labour Organization (ILO); viii) respect traditional land rights and local customs.

In our study, we evaluated the possibility of implementing a certification system, *Regional Model of Forest Certification*, for forestry exploitations of affiliated sites with an area less than 25 ha, in particular, populated by oak species suitable for use in cooperage (Vivas 2000) (Fig. 1). Galicia is an Autonomous Community of Spain that produces more than 8 million m^3 of timber, with the Galician forestry sector currently providing 12% of industrial employment. The contribution of the forestry sector is strategic because the Galician forest area is near to 2 million hectares, 69% of the total geographical area. However, the potential of Galicia as a timber producer can be considered

underdeveloped, since both the quantity and the unit value of forest production could increase considerably (Robak et al. 2012). One of the mainly causes for forest sector under-development is the high degree of private forest ownership in small, scattered exploitations. Private forests suppose more or less 97% of Galician forestlands, with about two thirds of those in plots of less than 2 hectares (frequently in several non-contiguous parcels). Approximately 30% of private forests are owned by communities, but despite this percentage, only several thousand hectares are sized to carry them out SFM (Ambrosio et al. 2003).

Oak forests, pure stands or mixed with other deciduous occupy an area of 246,445 ha, 18% of the Galician forestry area (MAGRAMA 2011). The most significant oak stands are found on abrupt slopes where have survived because felling would be very complicated (Ruiz de la Torre 1991). *Quercus robur* L. is the species that occupies a greater area, followed by *Quercus pyrenaica* Willd., and with scarce presence of *Quercus petraea* Liebl. The oak trees are little used in the forestry industry, the small plots and large number of owners doesn't facilitate its forestry, timber exploitation, and the later industrial development (Vivas 2000). Forest management of oak forests is virtually nonexistent and the use of oak wood is limited for firewood (Diaz-Maroto et al. 2005). This is the main reason why it would be well advised to develop a certification system for the SFM of the oaks for the obtaining of staves for the manufacture of barrels used in the whisky aging.

The certification process is available through the Galician Group of Forest Certification and Chain of Custody (CFCCGA), the main important certification institution in Galicia, and supported by the Government of Galicia, Spain. In the Fig. 2, shows the *General Scheme of Certification Process*. The certification system used by the CFCCGA is the Programme for the Endorsement of Forest Certification (PEFC). PEFC is a non-profit, non-governmental organization dedicated to promoting SFM by independent third-party certification (PEFC 2012). In all the membership plots of the system (plots that have received notification with the acceptance and with membership reference code), the harvesting should be carried out taking into account the mentioned below (Robak et al. 2012):

1) No delivery orders will be issued for certified timber plots whose date of registration in the application for harvest authorization is before the date of been membership of the system.
2) It's needed to submit a request for authorization by cadastral reference and properly complete all fields of the form. In addition to reflect all relevant observations, it is important to note the following data: i) complete cadastral reference; ii) total area of the plot to be harvested (ha), total wooded area in the forest (ha), and total area of the forest (ha); iii) target species exploited; iv) number of trees; v) volume of timber to be cut (m^3).

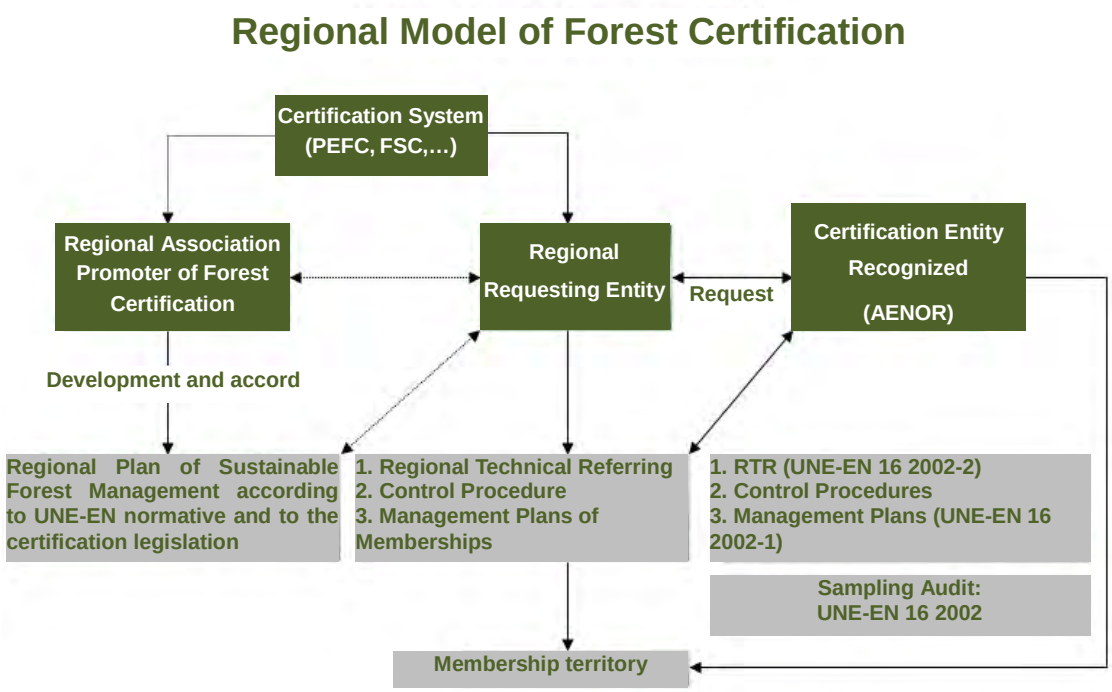

Fig. 1.
General scheme of Regional Model of Forest Certification.

3) Joined to the authorization of the Forestry Service, the exploitation must have all other legally required permits that would be applicable (water, patrimony, environment, etc.).
4) Companies engaged in harvesting plots must comply with the provisions in this regard in the Manual of Good Practice of the CFCCGA. Therefore, they must have sent the form *Commitment Compliance of the Good Practices Manual* signed and sealed by postal mail to CFCCGA before carrying out the first exploitation of a certified plot.

OBJECTIVE

Our research on the possibilities of implementing a certification system for sustainable forest management of small oak stands has as main objective the evaluation of the production of staves in these forests for the production of barrels used in whisky aging. Therefore, our goal was to assess this production, both economically and socially, because it could be a key factor of rural development for the scarce local population still lives in areas inhabited by oak forests.

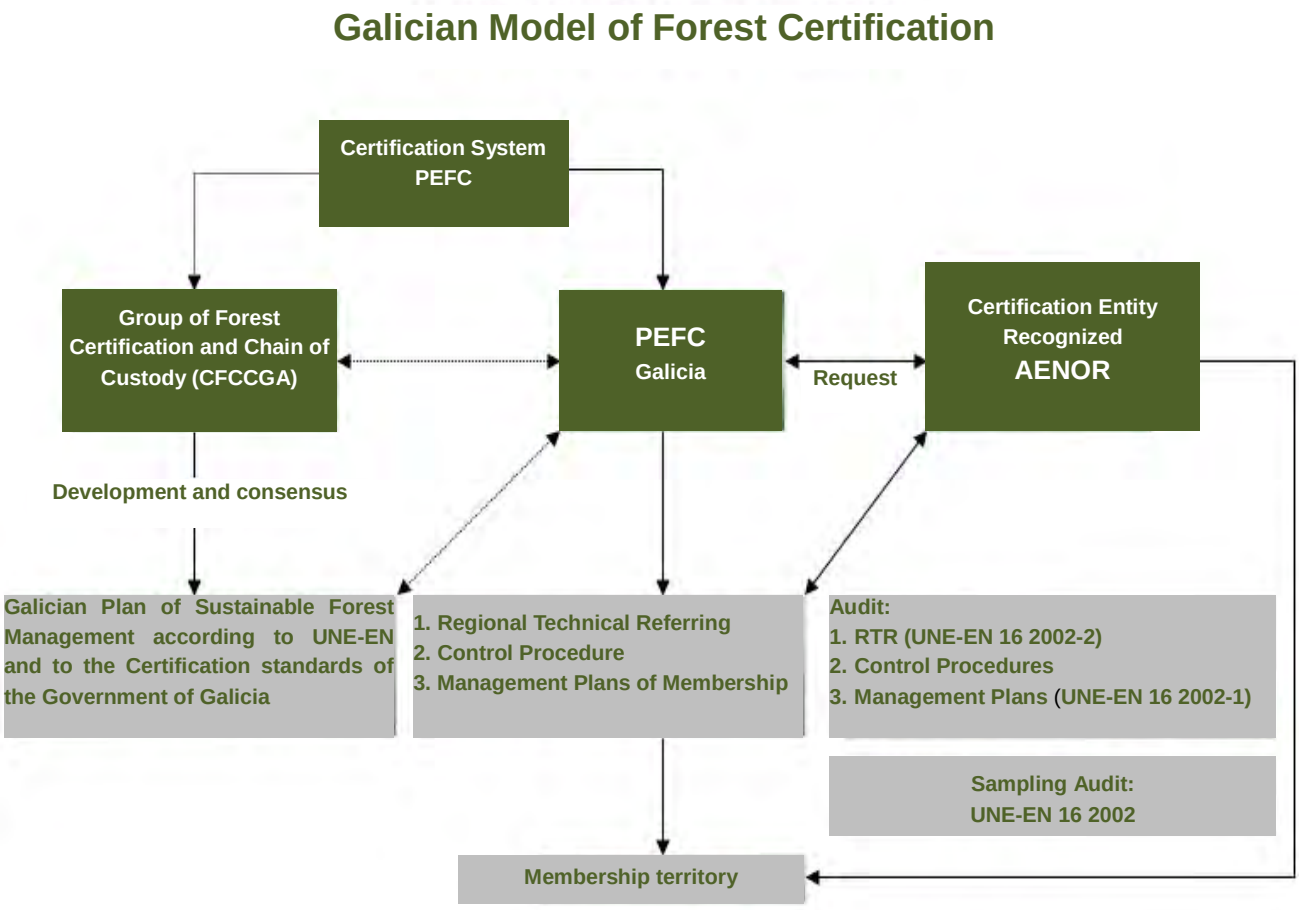

Fig. 2.
General scheme of Certification Process in Galicia.

MATERIAL AND DESCRIPTION OF THE METHOD

In the previous figure about the *Galician Model of Forest Certification* show as the Regional Requesting Entity (PEFC Galicia) needs a Management Plan for the membership forests. However, as the Group of Forest Certification and Chain of Custody (CFCCGA) have designed a Regional Certification Process, these plans are considered by great areas. In the Fig. 3 is described the territorial ambit of application of the joint management plans of the SFM System of the Group of Forest Certification and Chain of Custody (CFCCGA) in Galicia. As it can be seen, the joint management plans are divided in four different areas: North cost, Atlantic cost, central plateau, and the rest. Each area is also specified by a very important division of Forestry Management in Galicia: the District. Below, for example, municipalities of two districts of the province of Lugo, which has the largest forest area of the four Galician provinces, are listed:

Joint Management Plan Central Plateau (Lugo province)
District X – Denomination: *Terra Chá*: Abadín, Begonte, Castro de Rei, Cospeito, Guitiriz, Meira, Muras, A Pastoriza, Pol, Ribeira de Piquín, Riotorto, Vilalba and Xermade municipalities.
District IX – Denomination *Lugo-Sarria*: Antas de Ulla, Castroverde, O Corgo, Friol, Guntín, O Incio, Láncara, Lugo, Monterroso, Outeiro de Rei, Palas de Rei, Paradela, O Páramo, Portomarín, Rábade, Samos, Sarria, and Triacastela municipalities.

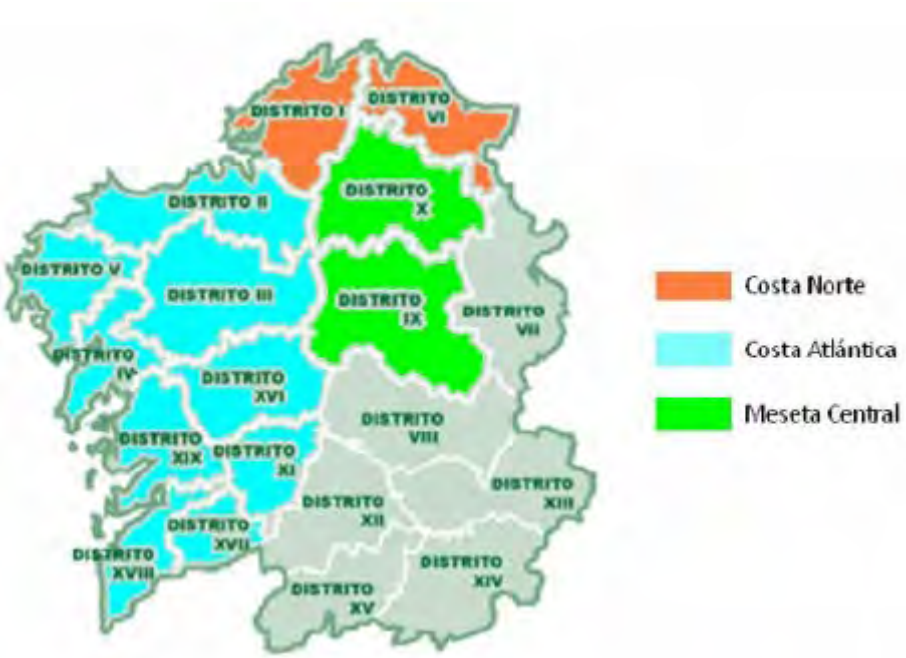

Fig. 3.
Territorial ambit of application of the Joint Management Plans of Forest Certification.

RESULTS AND DISCUSSION

Certification Process Timeline

1. Communication of the beginning of the exploitation

It can be done simultaneously for several plots.

• In any case, the date of harvesting beginning may be communicated to the CFCCGA prior to work start.

• This notification must be written including the reference code of each plot and the start date attaching the corresponding authorization for cutting.

2. Communication of the end of the exploitation and request of withdrawal note of certified timber

Also, it can be done simultaneously to several plots.

• Always the ending date of harvesting may be communicated to the CFCCGA. This notification must be in writing with the reference code and the ending date by plot.

• If withdrawal note certified timber is required shall be requested to issue to the CFCCGA. This request must be in writing and with indications if it's needed one or more invoices per plot and the desired mode for timber quantification. This quantification can be made with one of these two options:

- By **volume** indicated in the cutting permit.
- By **scale weighty**. The applicant should provide, for each reference code (or plot), the list of weights and the dates.

3. Forest Certification process taxes in Galicia

The Certification process has a direct cost of transaction. However, this cost can't be mixed with the cost of certificated timber. This is the cost of the process. The certified timber versus not certified timber may imply a price increase around 10 or 15%. Then, the current taxes for the emission of the withdrawal note are:

• 0.25 €/Tm (VAT not included): if the company that performs the harvesting in the membership plot is also member of FEARMAGA or Monte Industry (associations of timber manufacturing in Galicia).
• 2.00 €/Tm (VAT not included): in other cases.

4. Current situation of Forest Certification in the Galician private forests

The Galician private forests were under growing pressure to demonstrate SFM and due diligence with respect to legal source procurement. The increasing importance of certification in the forest product marketplace and legislative initiatives such as European Regulation (EU) No 995/2010 (European Parliament 2010) or Spanish Order Pre/116/2008 (Government of Spain 2008), have been instrumental in increasing pressure on the private sector. In addition, the global economic downturn that followed the collapse of major US financial institutions significantly reduced demand for forest products (UNECE/FAO, 2010). SFM, previously considered by many players as *a tool for reaching new markets*, suddenly turned into something *compulsory for maintaining declining core markets* (Robak et al. 2012).

Because of these increasing pressures, and other important factors such as the current growing demand from consumers for certified wood, forest certification systems have been applied successfully in the Spanish forestry sector and the Galician forestry sector, particularly.

CONCLUSIONS

Although in the early stages of its development seemed to progress quickly, the continued development and implementation of the SFM strategy framework has been delayed, with some major elements progressing only slowly. Greater time and effort spent obtaining clarity of roles and support from all stakeholders at the initial stages of the process, maybe slowing the early stages, would likely have led to more progress by this time.

Meanwhile, the forest industry in Galicia was facing increasing pressure by markets and public opinion to demonstrate that it had adopted sustainable practices. However, the Galician private forest sector, which is moving in this direction despite the difficulties caused by the small exploitations and fragmented ownership, has begun to realize that the full implementation of the SFM strategy framework, including the widespread application of different forest certification systems, could mitigate some of forest sector problems in this regard. Given that some positive results that link up very well with the administration's strategy framework have been achieved by private initiatives in a short period of time, there is reason to be optimistic that more and faster progress may be made now or in a short period of time.

ACKNOWLEDGEMENTS

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ASSESSING CORPORATE ECONOMIC DISTRESS: A STUDY OF THE WOOD CONSTRUCTION INDUSTRY

Fredrik LINDBLAD
Linnaeus University – Faculty of Technology, Institution of Mechanical Engineering
Address: Lückligsplats 1, 351 95 Växjö, Sweden
E-mail: fredrik.lindblad@lnu.se

Matti FLINKMAN
Ass.Prof.Dr. – Linnaeus University – Faculty of Technology, Institution of Mechanical Engineering
Address: Lückligsplats 1, 351 95 Växjö, Sweden
E-mail: matti.flinkman@lnu.se

Tobias SCHAUERTE
Ass.Prof.Dr. – Linnaeus University – Faculty of Technology, Institution of Mechanical Engineering
Address: Lückligsplats 1, 351 95 Växjö, Sweden
E-mail: tobias.schauerte@lnu.se

Abstract

Wood buildings are considered as a viable option to support the effort minimizing the current housing shortage in Sweden. Companies trying to develop into this industry are needed to increase the use of prefabricated wooden elements, volumes or modules in an industrialized way. Suitable companies to make this development could be found amongst firms producing wooden single-family houses. These companies currently act on a highly competitive market with many companies offering relatively homogeneous products or services. Therefore, differentiation towards the wooden multi-family house industry could be considered as a long-term strategy, minimizing the economic distress and improving the survival of the company.

The study is aiming at describing the development of economic distress and market concentration ratio in the Swedish industry for wooden single-family houses, for an eleven-year period from 2005 to 2015. The companies could be helped to understand, if and how the market concentration ratio and the economic distress are connected, linking company size to economic stability and efficient resource utilization. This will be conducted by applying Altman's Z'-score model, grouping firms into a risk, a grey or a safe zone, combined with calculating the industry structure by means of the concentration ratio model. The required data were collected from the annual reports of the 51 relevant firms in the industry.

Key words: *wooden single-family houses; industry structure; concentration ratio; economic distress; Z-score model; wooden multi-family houses.*

THE SWEDISH WOOD CONSTRUCTION INDUSTRY

The focus on finding green solutions have been an ambition of the EU, which have come to fruition in their development of the Europe 2020 strategy. The strategic aim is to create smart, sustainable economies for the citizens within the European Union (EU 2011, COM 2020). Hence, they have made recommendations to increase the use of wood in constructions linking it to enhanced focus on sustainable solutions. However, the EU has communicated no formal policies that specify the utilization of wood as a building material, which would be considered particularly crucial for the Swedish economy when increasing the building production during the next five years (FORMAS 2012; NRA 2012).

Sweden has been confronted with a high level of housing shortage during the past decade, which can develop into a problem regarding social unrest and segregation. This problem has been magnified by the continuous long-term increase of house prices in Sweden, which further enhances the problem since younger people with lower incomes have greater difficulties finding suitable accommodation. Furthermore, the general movement of people within Sweden can be restricted, which could have an effect on the general financial development in Sweden. According to the National Board of Housing, Building and Planning (Boverket) is it estimated that approximately 40 000 – 60 000 new housing units per annum are required to be constructed, to change this trend within the next five-year period (Boverket 2012). However, according to the Swedish National Trade Association for Wood and Furniture (TMF), the production during the period 2009 – 2015 was approximately 20 000 –

35 000 housing units per annum (TMF 2016a). Despite this development phase in comparison to the general production requirements of housing units, have an increase during the last years been noticed. Hence, the projected number for 2016 was estimated to between 35 000 – 40 000 housing units (TMF 2016b). However, the increased production of housing units is required to develop further if the projected demand during 2012 until 2025, with over 700 000 housing units shall be accomplished. The primary part of this development, 74%, is restricted geographically to the three big city areas, Malmö, Gothenburg and Stockholm (Boverket 2015).

The projected development supports a change from the traditional building materials towards a greater focus on wood solutions. Currently, concrete makes up 89% of the building material used within the Swedish industry producing multi-family houses, whereas wood solutions only constitute close to 9% of the market (TMF 2016a). Therefore, the positive environmental benefits should also be considered when evaluating wood as a suitable construction material for multi-family houses. Thus, enabling the increased development of sustainable building solutions for multi-family houses in Sweden (Nord and Widmark 2010; Schauerte et al. 2014).

Besides the environmental aspects, a benefit utilizing wood in the construction of multi-family houses is the development of onsite assembly methods. The investigated companies are well familiar with this production and assembly process since they have utilized this methodology for a long time (Schauerte 2010). Furthermore, it has also been indicated to be more beneficial regarding cost savings, quality, work environment and logistics than those related to onsite construction (Stehn and Brege 2007; Mahapatra and Gustafsson 2008).

Despite these benefits, as displayed by producers of wooden single-family houses, currently, a limited number of companies within the industry for wooden multi-family houses take full advantage of the possibilities associated with a higher degree of prefabrication (Stehn and Brege 2007). This could be an explanation for the problems related to inefficiency, relatively low productivity and increased production costs within the industry (Schauerte et al. 2013). Therefore, companies producing single-family houses are required to deal with these issues if they successfully shall establish themselves producing wooden multi-family house.

The problems associated with diminishing productivity and profitability are not only related to an inefficient production but also to shifting market conditions. According to TMF (2016b), from 2007 to 2012, the number of finalized wooden single-family houses in Sweden decreased from about 12 100 units to 4 800 units per annum. Furthermore, the existing production methodology and structure has also led to increased production costs and a low degree of resource utilization. The production costs/m^2 have increased from 16 258 SEK in 2001 to 30 988 SEK in 2015 (SCB 2017), i.e. an increase of around 91 % in nominal value, as described in Table 1.

Table 1

Development of production cost/m^2

Year	**2001**	**2002**	**2003**	**2004**	**2005**
Production cost/m2	16 258 kr	16 691 kr	17 966 kr	19 198 kr	19 684 kr
% change (based on 2001)		2,7%	10,5%	18,1%	21,1%
Year	**2006**	**2007**	**2008**	**2009**	**2010**
Production cost/m2	20 484 kr	22 985 kr	25 107 kr	24 745 kr	26 011 kr
% change (based on 2001)	26,0%	41,4%	54,4%	52,2%	60,0%
Year	**2011**	**2012**	**2013**	**2014**	**2015**
Production cost/m2	28 221 kr	27 042 kr	28 747 kr	31 064 kr	30 988 kr
% change (based on 2001)	73,6%	66,3%	76,8%	91,1%	90,6%

For companies producing wooden single-family houses to differentiate the product and develop into the market for multi-family houses is however connected with investments and risk taking. Also, there is a requirement for production efficiency to improve the competitiveness and profitability of the company (Besanko et al. 2013; Bottazzi et al. 2008). However, many producers of wooden single-family houses may face profitability issues, resulting in financial problems, while converting to new production systems accommodating multi-family building solutions.

OBJECTIVE

The main purpose of this study is to investigate if any connection exists between the corporate economic distress and the concentration ratio of Swedish companies producing wooden single-family houses. Here, two objectives are identified. First, by using the latest available economic figures conduct an evaluation of the corporate economic distress over a period of some years, which will reveal insights into existing trends or tendencies. Secondly, an assessment of the market structure by evaluating the concentration ratio of the companies included in the study, which will provide an understanding of the market diversification.

Hence, it is important to assess the participating companies' potential to make investments over time and if the concentration ratio affects the company ability for investments and long-term efficient resource utilization.

THEORETICAL BASE

Industries are constantly developing and changing in character, which creates challenges that need to be addressed by the companies, determined by the specific situation for that industry. Therefore, having the ability to generate a comprehensive understanding of the industry, based on an in-depth understanding of the market structure, provides necessary support when developing a suitable company strategy. This is commonly measured trough the industry concentration, which can be described as the degree of concentration relating to the output of all firms in that industry (Rhoades 1993), or more commonly mentioned as the concentration of companies. The industry concentration can be measured by identifying the companies' relative position on the market, also known as market share (Besanko et al. 2013). One of the most commonly used methods to measure market structure or market concentrations is the company deposit Concentration Ration (CR_n) (Al-Muharrami and Matthews 2009).

The market structure model identifies the level of concentration on a specific market or industry, by understanding the Concentration Ratios (CR_n) between the companies on the market. The market concentration is calculated by the total sum of turnover for all companies within a market or industry, in combination with the turnover of the company (Matthes and Poetzsch 2002), see Equation (1):

$$CR_n = \sum_{i=1}^{n} x_i \quad (1)$$

where: the CR_n describes the n largest firms competing on the specific market, generating x_i market share (%) for these companies(Matthes and Poetzsch 2002). The maximum value for CR_n is 100%, which suggests a very dominant market position comparable to a monopoly situation.

The German Federal Cartel Office (2013) have identified three levels of concentration ratios, stating that companies on a market are presumed to be dominant if one company exceeds 33.3%, or if three or fewer firms, reaching a combined market share of 50.0%, or if the market consists of five or fewer firms reaching a combined market share of 66.7%.

There are several different methods available to measure corporate economic distress. However, according to Crouhy et al. (2001), four models are most commonly applied: models based on discriminant analysis, linear probability models, probit models and logit models. The goal of these models is to find ways to predict bankruptcy, which has been the focus of many studies conducted by different authors, such as Edmister (1972), Shumway (2001), Hillegeist et al. (2004) and Elliott et al. (2014). However, recently Altman's Z-score model have been used more extensively by researchers and financial analysts due to its prediction accuracy (Elliot et al. 2014 and Gunathilaka 2014). The Z-score model is based on multiple linear discriminant analysis, which could indicate that a firm will turn bankrupt within two years and the accuracy of these forecasts was varying between 75 % and 90 % (Altman et al. 2014).

Altman's original Z-score model included five variables, which were chosen based on their contribution to prediction accuracy and their inter-correlation (Altman 1968). However, this model was developed for publically listed companies and had to be further developed to allow for private industry companies to be included as well. Adjusting the old model to the new Z'-score, Altman replaced variables towards a more suitable approach for private firms. The resulting model is shown in Equation (2) (Altman 1983):

$$Z' = 0.717X_1 + 0.847X_2 + 3.107X_3 + 0.42X_4 + 0.998X_5 \quad (2)$$

where: these five ratios belong to different key economic classifications, i.e. liquidity, profitability, leverage, solvency and activity and can be interpreted as follows:

X_1: working capital/total assets. Working capital is calculated as current assets minus current liabilities. Total assets include all assets on the balance sheet. This ratio describes company's liquidity related to its size and its ability to meet short-term debts (Al-Rawi et al. 2008).

X_2: retained earnings/total assets. Measuring retained earnings gives a picture of what actions companies have taken regarding its profits. This ratio reflects a company's aggregate profitability over time since research showed that the risk for failure of companies is related to the age of the company (Dun & Bradstreet 1994, Eidleman 1995).

X_3: EBIT/total assets. EBIT measures profitability or earnings before interests and taxes. This ratio reflects a firm's earning power of its assets excluding the one-time effect of interest and taxation (Muthukumar and Sekar 2014).

X_4: book value equity/total liabilities. The book value of all assets is measured in relation to the total amount of company debts. If the equity book value of a company is less than its total liabilities, a firm can become insolvent in the short run and bankrupt in the long run (Taurell and Augustsson 2012).

X_5: sales/total assets. This measures the asset's sales generating capacity, also referred to as the manufacturing capacity of the company's assets (Taurell and Augustsson 2012) or more commonly the management's capability to compete on the market (Muthukumar and Sekar 2014, Altman et al. 2014).

Thereafter, the calculated values for the independent variables X_1 to X_5 are multiplied with the respective discriminant coefficients according to Equation (2). The resulting Z'-score is interpreted according to pre-established cut-off scores or zones, as presented in Figure 1.

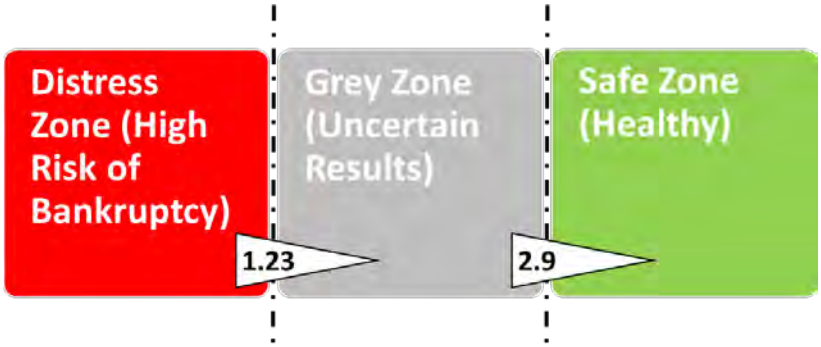

Fig. 1.
Z'-Score Classification Areas and cut-off levels (Altman et al. 2013).

Figure 1 display three classification zones with their respective cut-off scores. If a company present a Z'-score below 1.23 indicates that the company is in the distress zone and probably will face bankruptcy, unless appropriate actions are taken by the management within the company. However, a company that displays a Z'-score between 1.23 and 2.9 find themselves in a grey zone, i.e. a relatively unpredictable situation based on the financial results. Finally, Z'-scores of 2.9 or higher indicate that companies' financial situations can be regarded as healthy. These companies face a situation with minor economic risk and have promising potential for future development (Altman 2001). These categories, as presented in the Z'-score model can function as support during the development of investment strategies or market positioning activities for the individual company.

DATA COLLECTION

The companies included in this study were required to be producers of wooden single-family houses and to be located in Sweden. The selection process was initially initiated through an online statistical database for Swedish wooden single-family houses. The resulting list of companies was further edited by removing companies that were too small regarding employees. As a cut-off level, ten employees were chosen. This resulted in a list of 51 companies. Different ownership models between these companies were not considered.

Since sensitive competitive relationships exist between these companies, disclosing economic positions of the specific companies could be used for competitive actions on the market. Therefore, no names of firms are revealed in this study and the companies are handled as anonymous units of analysis.

For the chosen 51 companies, all necessary economic data to calculate the Z'-score and the CR_n according to Equation (1) and (2) was collected from their balance sheets for the years 2005 to 2015. This eleven-year period was chosen since balance sheets for that period were publically available in an online database at the time.

RESULTS AND DISCUSSION

In order to highlight the development of economic distress and market concentration ratios and their possible interdependencies.The Altman's Z'-score model and concentration ratio approach were used, accomplished by grouping the companies into concentration ratio groups. Firstly, the CR_1 group consisted of companies with the largest market share, the CR_3 group including companies with the three largest market shares and finally the CR_5 group with firms having the five largest market shares. Besides a descriptive analysis, a simple regression analysis based on Fixed Effect model was carried out.

As shown in figure 2 the Altman's Z'-score value (total average) of 51 enterprises remains above Altman's High score, except for two years in the middle of the period, the average being 3,6. The last years indicate an increase towards the levels at the beginning of the measured period.

As to Z' scores of CR_1, CR_3 and CR_5 their average value over the period do not significantly differ from the sample average. The overall impression is that they fluctuate around the mean Z' score value, except Z' score of CR_1, which shows a remarkable variation and clearly a decreasing trend, but still remaining within Altman's grey uncertain zone towards the end of the period. The Z' score values for the CR_3 and CR_5 groupings tend to slightly decrease but remain around Altman's High score limit towards the end of the period.

One reason for higher Z' score values of the CR_3 and CR_5 groupings should be, among others, the fact that these (average values) consist of underlying companies' Z' score values. By scrutinizing instead individual Z' scores of, e.g. the five largest companies highlights clearly that the annual amplitudes over the period are quite large and for example the Z' score values of CR_3 and CR_5 (here as single companies' individual values) reach Z' scores within the distress zone in some few occasions; i.e. below Altman's Low score value (Figure 2). For these five companies their market shares; i.e., concentration ratios have remained relatively stable over the period resulting in the same rank of market shares in-between the companies.

Thus, it turns out that the development of economic distress and market share (market concentration ratio, CR) do not indicate any drastic changes/movements from 2005 to 2015 among companies constituting CR_1, CR_3 and CR_5 groupings and their Z' score values. Possibly one can notice a slightly decreasing trend of Z' scores including some recovery at the end of the period. Also, the enterprises forming CR_1, CR_3 and CR_5 groupings seem to be stationary; i.e. keeping their CR_n ranking, respectively.

In order to further explain eventual relation (dependency) of Z' scores of market shares (concentration ratio concept) a simple regression analysis accomplishing the Fixed Effect model on panel data was carried out. By selecting this approach, the number of observations could be increased for the purposes of regression analysis. The outcome of the regressions was that applying a so-called individual effects model approach the estimated model could explain 27 % (adj. R^2) of variation in Z' scores. Regardless of the low explanatory power in the estimated model Z' = 1.987 + 0.489***CR the concentration ratio (CR) turned out to be a significant explanatory variable indicating that a unit change of CR would lead to an average 0,489 increase of Z' score value.

Still another fixed effect model approach exploring time effects resulted in the equation Z' = 0.578 + 0.212CR** where CR again turned out to be a significant explanatory variable though with somewhat lower significancy compared with the previous one. As well the explanatory power was low, only 11 % of the variation in Z' scores.

A combination of individual and time effects model gives the equation Z' = 2.074 + 0.676**CR with an explanatory power of 20 % and the CR being a significant explanatory variable.

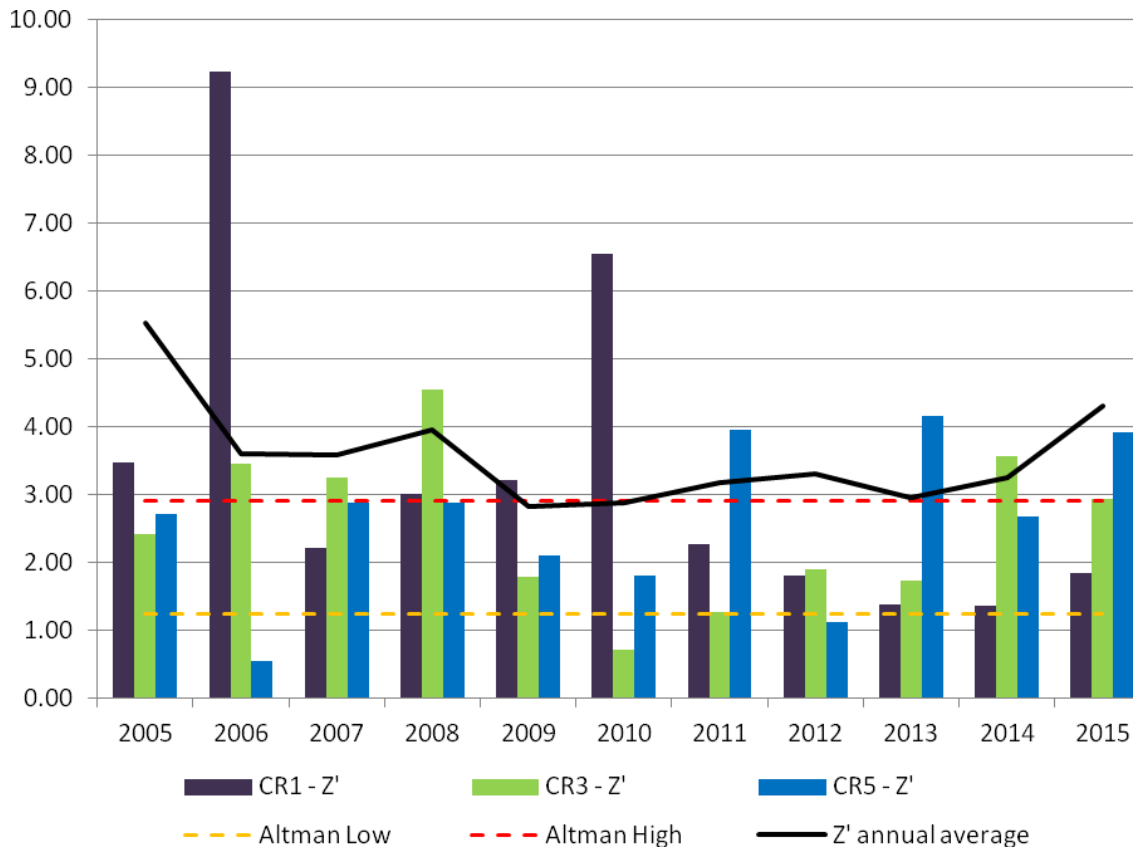

Fig. 2.
Altman's Z' scores for groupings CR_1, CR_3, CR_5 and the average Z' value for the total sample of 51 companies, including Altman High and Low limits.

CONCLUSIONS

The aim of this study was to investigate if any correlation exists between the market concentration ratios of the CR_{1-5} companies and an economic distress model for the Swedish companies producing wooden single-family houses. This was conducted using economic figures derived from the companies' annual reports over an eleven-year period. Thereby reveal if any existing trends or correlations exist. Also, provide insight regarding company size, and its possible effect on the economic situation. This could have a contributing effect on companies' ability to make suitable investments, market development activities and product development towards the wooden multi-family house industry, in comparison to companies with smaller market shares.

However, the result has not been able to clearly demonstrate any correlation between the concentration ratio model and the economic distress model demonstrated by Altman's Z'-scores. Despite any clear link between the market share of the company and its financial health, significant explanatory values have been established for then various models independently, even if the significant explanatory power have been low. Hence, a reason for the uncertainty demonstrated when reviewing the correlation between the models, and the significance of each model independently could be derived from the recent financial recession. This period has provided a financially uncertain situation for main companies, forcing them to take actions in line with a financially challenging situation i.e. depleting its equity and making limited developments of fixed assets due to general financial constraints.

However, the result shows that those companies with a large market share, as demonstrated by the CR_{1-5} companies, have greater ability to absorb a long financial recession due to its initial high equity levels and fixed assets, compared to companies with a smaller market share. Therefore, the general financial situation and the strength demonstrated by the market share for the largest five companies included into the study generates a fairly good potential for these companies to invest in production development and product development, supporting an entry to the segment for wooden multi-family houses.

Further research can offer comprehensive information about how the equity and fixed asset levels within companies producing wooden single-family houses can sustain and support long-term financial recession and how these are linked to the companies' ability to provide a stable revenue.

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IDENTIFYING DRIVERS FACILITATING PRODUCT DEVELOPMENT WITHIN THE INDUSTRY FOR WOODEN MULTI-FAMILY HOUSES

Fredrik LINDBLAD
Linnaeus University – Faculty of Technology, Institution of Mechanical Engineering
Address: Lückligsplats 1, 351 95 Växjö, Sweden
E-mail: fredrik.lindblad@lnu.se

Tobias SCHAUERTE
Ass.Prof.Dr. – Linnaeus University – Faculty of Technology, Institution of Mechanical Engineering
Address: Lückligsplats 1, 351 95 Växjö, Sweden
E-mail: tobias.schauerte@lnu.se

Abstract

Sweden is forecasted to grow its population with 1.1 million people over the next eight-year period, increasing the demand on the construction phase of housing units throughout Sweden. However, at present, 240 of Sweden's 290 municipalities show an existing deficit of available housing units in their regions, resulting in inherent difficulties fulfilling this demand utilizing the current production structure. Therefore, further utilizing wood as a building material could contribute to minimize the gap, as well as fulfilling the EU's goals towards the Europe 2020 strategy and the EU forest strategies, focusing on development towards innovation, bio-economy, sustainable sourcing and use of raw materials. This study is aiming to identify drivers supporting the Swedish industry of wooden multi-family houses to enable market growth through competitive and sustainable strategies. The representatives within the building process identify drivers, how they perceive their effect on the companies' abilities to develop based on long-term and short-term strategic impact. Thus, the goal is to find ways in which wooden multi-family houses could compete as a building solution, compared to established solutions, thereby increasing the market share in Sweden. The methods used in this study is surveys distributed to representatives from municipalities, developers, contractors, architects and real estate companies.

The result identifies three change drivers influencing the industry development for wooden multi-family houses in Sweden: technological-, knowledge- and environmental- drivers. These drivers have an effect on the companies' ability for successful new product development and for development of sustainable strategies towards market growth for wooden multi-family houses.

Key words: *industry drivers; wooden multi-family houses; sustainable development; competitive advantage; new product development.*

INTRODUCTION

According to forecasts, the population of Sweden is expected to increase by approximately 1.1 million people over the next eight years. During the period 2012 – 2015, approximately 127 700 homes were constructed and the population of Sweden grew with 368 200 people during the same time-period (Eriksson 2016). The task for planning a suitable construction rate in response to the growing population falls partly on the municipalities in Sweden. However, an investigation conducted by Boverket (National Board of Housing, Building and Planning) during 2015 estimated that approximately 240 of Sweden's 290 municipalities show an existing deficit of available housing units in their regions (Boverket 2016).

According to Boverket, forecasted demand for construction in Sweden is approximately 710 000 housing units to be built during 2015 – 2025 (Boverket 2015). By 2020, the industry needs to build 88 000 housing units per year to meet the population growth. Further, additional production volume is required to compensate for the building shortage from previous years, based on insufficient production pace in comparison to market demand. This poses a challenge for the building industry considering its relatively low output during the past decade, with an average annually output from 2007 until 2015 of 14 803 apartments. The production volume has increased throughout this period, during 2015; 23 916 apartments were constructed out of concrete and 2 322 apartments were built using a wood solution (TMF 2017). Development strategies to fulfill the construction requirement until 2025 be to further explore wood as a suitable construction material and to investigate the development drivers for producers of multi-family houses in wood. This poses an additional challenge, since the requirement for building housing units will be at a high level in the foreseeable future. Hence, the

additional need for sustainable production technology at all levels of the value chain is required, thereby improving the production output (Eriksson 2016).

Although, the output of housing units has to increase greatly during the upcoming years, the work against climate change will have to continue combined with a demand to develop building techniques in line with a sustainable economy (FORMAS 2012; NRA 2012). This relates to strategic advances influencing wood-based industries, highlighting the importance for the EU to enhance investments in green building solutions to comply with the climate and environmental targets. Therefore, the EU has recommended the use of wood as a sustainable building material (EU 2012. COM 433).

The development towards a long-term sustainable building industry of wooden multi-family houses requires a massive build-up of the industry to fulfill the expected development. This can not be achieved without the understanding of the existing drivers within the industry and the understanding that these might shift as the industry evolves. According to Björheden (2006), effect and importance of different drivers vary depending on the type of company, the geographic scale and the operational environment within the industry. Thus, drivers may have very different effects within a country, where regional and local conditions may vary, creating specific industry drivers to take into consideration. Furthermore, political decisions are perceived as important drivers for the development, specifically when expressed through legislation, duties, and taxation. In addition, other general drivers are considered to be areas such as social, economical and institutional drivers, identified as influencing the market development and new product development (Björheden 2006; Tudor et al. 2006).

New product development (NPD) is an area of importance for the development of an industry and individual companies, which is affected by internal and external drivers. NPD is considered as strategically important for companies having the ambition to develop their market share based on product usability and quality (Ciappei and Simoni 2005; Johne and Snelson 1990). This further enhances the dependence of product innovation and business success on industry drivers and the ability for companies to gain competitive advantage through successful NPD (Hassanien & Dale 2012). However, according to Chiu and Yong (2004) and Roberts (2004), NPD should be supported by several company activities to generate synergies trough drivers, such as financial planning, a multi-stakeholder team, governmental activities, the community and expert advisors, thereby maximizing the possible outcome from NPD and a stronger market position.

Despite the importance of NPD for company development, the primary activity for the companies will be towards aligning the company strategy in regard to its context, i.e. the external and internal environment of which the company is involved in, including competences and resources. Actively working within these areas of the company maximize the effect of industry drivers, which have proven to have important implications towards the company's performance (Venkatraman 1989; Venkatraman and Prescott 1990; Anderson and Zeithaml 1984; Bourgeois 1981).

OBJECTIVE

The purpose of this study is to investigate existent development drivers for the industry producing wooden multi-family houses in Sweden. Firstly by generating an understanding of the perception by the main stakeholders within the building process regarding the drivers influencing the industry. Secondly, evaluating the result trough the driver's impact on companies from a strategic perspective. This analysis will be useful for the industry or individual companies developing sustainable strategies through NPD, increasing their market share in comparison to traditional building materials.

THEORETICAL FRAMEWORK

Creating possibilities for companies to successfully develop within an industry is based on several factors. Aladwani (2001) discussed the importance of drivers for business development, mentioning competitive position, consumer demands, distribution channels and business image as important drivers for company development. Therefore, companies try to adjust towards drivers, reacting to external and internal challenges influencing their growth, by optimizing the combined effect of resources and drivers.

The resource-based view (RBV) provides an understanding of how strategic activities create an competitive advantage on a company level, by clarifying strengths and capabilities. According to Wernfelt (1984), companies organize their resources, in combination with market conditions, in a way making their products and services hard to imitate and thereby generating sustainable competitive advantage. Kim and Park (2006) suggested that two factors make a company competitive, product quality and brand, which in combination with good design, innovative products, and brand building support companies in achieving a competitive advantage. However, Hax and Wild (2002) discuss that

competitive advantage, based on RBV, is factor driven, i.e. dependent on a company's development of resources and its capabilities in comparison to market conditions and drivers.

Having the ability to utilize internal resources and competences within a company, combined with existent market drivers, facilitates innovation and improves competitive advantage (Chesbrough 2003). This has also been discussed by Prahalad and Hamel (1990) and Pavitt (1990), highlighting the importance of efficient usage of internal competences in combination with external drivers facilitating NPD. Defining NPD provides a broad span of activities, from new-to-the-world to minor revisions of existing products, where many development activities can occur, e.g. Research and Development (R&D) and technological breakthrough (Majava et al. 2013).

The ambition for companies is still to find ways to lower costs throughout the value chain, delivering the required service levels and quality. Optimizing the influence of drivers accomplishing higher profits is still considered as a competitive strategy for companies. Therefore, focus on achieving economies of scale and low-cost production will continue. However flexibility and innovation will continue to be an important aspect for companies striving towards strengthening their competitive position (Ülkü et al. 2005). Based on the scope of this study, three main categories of drivers have been identified; technological-, knowledge-, and environmental drivers. Efficiently combining internal resources with these industry drivers facilitates NPD. These drivers can be unified under a broader concept enabling company growth, discussed by John et al. (2001) as change drivers. Figure 1 below describes the various change drivers influence on industry structure and company activity.

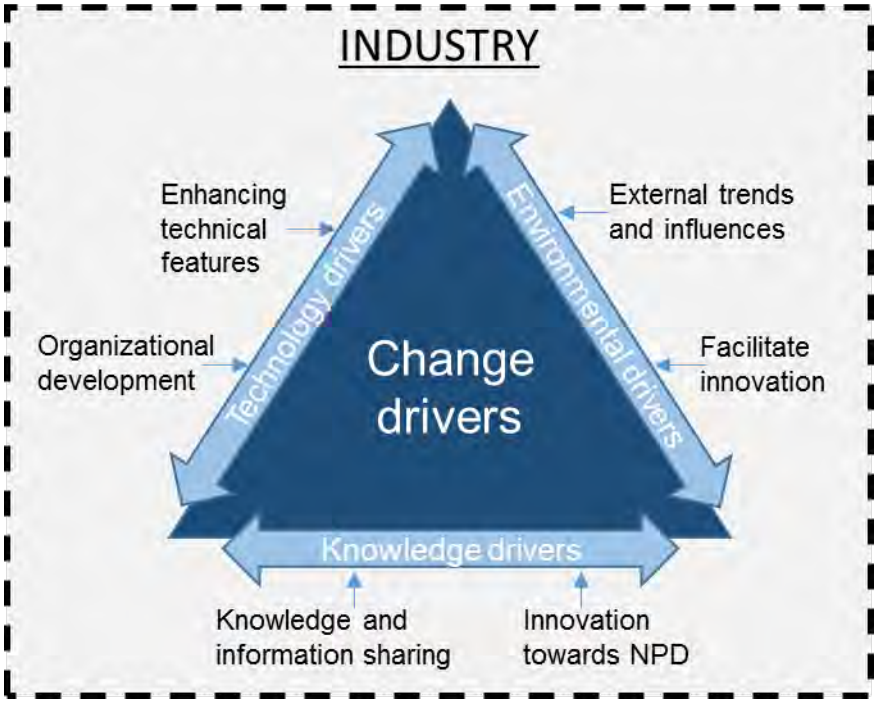

Fig. 1.
Industry change drivers influence on company activities.

The first category, technological drivers, consists of activities associated with enhancing technical features, enabling organizations to develop products and processes to create a competitive advantage. Technological capabilities are for many companies considered as the main driver towards successful product development (Gann and Salter 2000; Mitropoulos and Tatum 2000; Verona 1999). Furthermore, it is important to coordinate the technological development by using official programs or institutions as platforms, thereby facilitating development based on industry requirements and capabilities. Miozzo and Dewick (2002) discussed the importance of long-term relations between companies and external knowledge centers providing access to new technologies in the construction industry, which further enhance the possibility for the industry to develop, based on the efficient use of existent drivers.

The second identified category is knowledge drivers. This includes various activities required for companies to enable knowledge and information sharing between organizations and institutes that are associated with innovation towards NPD (Goverse et al. 2001). A study conducted by Toole (1998) identified that a company's ability to accumulate and process information about new advances in

technology contributed to the company's possibility towards successful innovation and NPD. Further, Toole (1998) and Veshosky (1998) discussed the positive effect of information gathering and knowledge sharing as an enabler towards successful innovation and development.

The third category, environmental drivers, has a relatively broad scope including general trends and influences from market, society, and competition that encourage organizations to innovate (Toole 1998; Gann and Salter 2000). A study conducted by Arditi et al. (1997) found that the innovation rate increased, based on general market forces pushing for product development. In this context, public policy instruments have the possibility to drive development in a specific direction and studies by Seaden and Manseau (2001) highlight the positive effect of innovation and change when governmental organizations were in the position of clients in construction projects.

Furthermore, a study of the Dutch construction industry concluded that development of regulations was required to stimulate the use of wood within the construction industry in order to stimulate innovation and NPD. This, according to Goverse et al. (2001), could be various regulatory measures by the government. However, Björheden (2006) displayed in his study of the Swedish market that policy is not enough. The political system must publically support and contribute to the development of a stable market situation. This was further discussed by Markström et al. (2016), mentioning that local strategies by municipalities for construction of wood buildings act as an incentive for the industry. Thereby providing necessary drivers for companies to invest towards innovation and NPD, possibly strengthening their competitive advantage.

METHOD & DATA

The research conducted in this study is based on a specific wooden multi-family house building project in Sweden. Data collection was predominantly focused on companies that are actively involved in the process of that project. It is a method consistent with the convenience sample strategy of data collection, finding suitable respondents to be included in a study. This method was used to find respondents within the identified building process (Zikmund 1997).

To start with, drivers for market development were identified from existing literature. By means of senior-level managers from the industry, these drivers were validated and selected, with respect to the context of the study. The resulting questionnaire was sent to 157 respondents via an online survey. To gain a broad understanding of the perception of drivers, respondents from municipalities, developers, contractors, architects and real estate companies were approached.

The survey took place during autumn 2016. The response rate on the survey was approximately 40%. In the survey, the respondents were asked to evaluate the importance of the questions, offering a comprehensive picture from procuring or commissioning a building, through construction and finally the operational use of the building. This provides an end-to-end perspective. Focus was on three main segments: company information, market information and market drivers. Each segment had several subsections to emphasize the context of the questions.

Considering focus was on companies producing multi-family solutions, data was analyzed in relation to those companies only producing wooden multi-family houses. This provided a 31% response rate. These companies are not only involved in the construction of multi-family buildings but also other building solutions i.e. single-family houses, public buildings and in other types of construction projects. In addition, 91% of the companies build higher than 3 floors and 54% build higher than 5 floors out of wood. Further, some companies are also active in several different roles within the building process i.e. procurer, developer, architects, contractor, sub-contractor and real estate company, providing a comprehensive value chain perspective.

The survey consisted of 18 main questions and 14 sub-questions designed to use a 10-graded Likert scale. However, some question were open-ended, which allowed the respondents to elaborate on specific industry information. Further, some questions had a *yes* and *no* option, intended to be a filter for some of the following questions.

Researchers analyzed the responses and established possible trends. Further, data was analyzed quantitatively for those questions with an open-end response option. Based on an analytical approach, focus was to identify drivers enabling the development of the market for wooden multi-family houses in Sweden.

The data from the survey was structured combining the responses from the participants with the appropriate change drivers, displayed in Table 1 below. The table is based on 18 main questions and 14 sub-questions, where the average result from the question in the survey has been classified into three levels based on importance, thereby providing the ability to classify the different drivers. The evaluation of question 7 and 9 was incorporated into the three-graded scale in Table 1, based on the percentage, i.e. if > 67.3% answers *Yes*; equals major importance. Question 3 is based on the same structure i.e. if > 67.3% spend less than 5% is perceived as being of minor importance.

Technology is the first driver, consisting of six questions including topics such as R&D, economies of scale and product differentiation. Question number one was of most importance for the respondents providing an average score of 7.19, positioning it as being of major importance for the development of the industry. On the other hand, question number three, relating to the extent the companies reinvest in R & D based on their production cost, was perceived as being of less importance from the respondents. The response showed that 78.7% of the respondents only allocated less than 5% of the production cost towards R & D. Yet, of the respondents, 45.8% considered the R & D cost as a limitation towards technological development for the industry.

Table 1

Review of industry change drivers

			0 - 3,3	3,4 - 6,6	6,7 - 10
Change Drivers			***Importance***		
Technological drivers	*Score*		*Minor*	*Medium*	*Major*
1. Are economies of scale a necessity, and will this influence the development of wood buildings	7,19				X
2. What significance do you put on research and development within your company	6,25			X	
3. What proportion of the production cost is connected to R & D	< 5% = 78,7%	6-10% = 21,3%	X		
4. Is cost for R & D considered a limitation towards technological development	Yes = 45,8%	No = 54,2%		X	
5. To what degree is product differentiation important for the development of the wood building industry	6,12			X	
6. What is the importance of product differentiation for the development of your company	6,11			X	
Knowledge drivers					
7. Do you continually work to create competitive advantage for your company	Yes = 89,1%	No = 10,9%			X
8. What kind of business intelligence activities do you conduct prior to an investment	Based on 6 sub-questions				
Complie information	23,6%				
Conduct a market analysis	21,4%				
Identify sales channels	12,1%				
Analysis of production capacity	20,7%				
Review alternative solutions	20,7%				
Misc	1,4%				
9. Have you got financial possibilities to invest towards strengthening your competitive possition within your industry	Yes = 81,2%	No = 18,8%			X
10. What kind of advantages exist regarding investment possibilities	Based on 8 sub-questions				
Stable political situation	12,3%				
Market size	16,4%				
Growth opportunities	25,3%				
Good infrastructure	14,4%				
Possibilities finding partners in the Value Chain	9,6%				
Access to personnel	8,9%				
Stable financial situation	12,3%				
Misc	0,7%				
11. Are you required to make investments to optimize your competitive position	Yes = 48,9%	No = 51,1%		X	
12. Is an improved collaboration between the participants in your value chain a necessity for improved competitiveness of wood buildings	7,02				X
13. Are you able to identify possibilities to achieve economies of scale and how significant is this for your company	5,92			X	
Environmental drivers					
14. Market strength of concrete	8,17				X
15. Insufficient understanding of wood as a building material	6,67				X
16. Are there any rules or regulation that limits competition	Yes = 62,5%	No = 37,5%		X	
17. Essential for legislation to change	6,23			X	
18. To what degree can you change rules or legislation	6,23			X	

Knowledge drivers are the second category, including 7 main questions and 14 sub-questions. This category includes topics such as competitive advantage, business intelligence, investment decisions and the possibility or importance to achieve economies of scale. Question seven received the highest attention with 89.1% of the respondents continually working to strengthen their market position by e.g. participation in research projects, project development and internal project teams focusing on development issues. However, only 48.9% consider it a requirement to invest optimizing

their competitive position, which indicates that current resources suffice. Both question 8 and 10 have been answered by 100% of the respondents showing the value of information prior to investments decisions and the importance of a healthy business climate supporting investment activities. Out of the sub-questions are the most important to collate information, market analysis, production capacity and general growth opportunities those questions that receive most responses. Further, linked to question 10, requirements for municipalities to promote wood-buildings for new construction projects were of importance, rather than utilizing traditional building materials such as concrete.

The final category, environmental drivers is based on five questions related to external environmental factors influencing the development of wood as a suitable building material for wooden multi-family houses. The main identified issue influencing the industry development is the strength of traditional building materials, predominantly concrete, which received an average score of 8.17. It makes this question the most significant, by the respondents, out of all questions asked in the survey. Furthermore, the perception that rules and regulations limit the possibilities to compete is shared by 62.5% of the respondents making this an important focus area. Hence, the respondents feel a requirement to change the legislation to improve their abilities to compete. However, they don't consider themselves having the possibility to do so, which provides an average score of 6.23, positioning these questions close to being of major importance for the industry. In addition, several of the respondents had supplementary comments regarding the environmental driver e.g. requirement to introduce taxation on CO_2 emissions, demand on a life cycle analysis, stopping the spiraling cost increase within the industry and a faster phase changing regulation to support sustainable building solutions.

DISCUSSION

Meeting the main objective of this study, to investigate existent development drivers for the industry producing wooden multi-family houses in Sweden, was fulfilled. This was conducted by mapping the industry perception regarding main drivers influencing the development of the industry, which generated a structure consistent with three categories of drivers: technological-, knowledge-, and environmental- drivers. Thereafter, the second objective was to provide a structure demonstrating the impact of the identified drivers, which was conducted by grading the responses. This generated an overview of what drivers that were perceived to be of greatest importance for NPD and industry development. The result is summarized in Table 2.

Table 2

Classification of industry change drivers

	Minor	***Medium***	***Major***
Technological drivers	17%	67%	17%
Knowledge drivers	0%	29%	71%
Environmental drivers	0%	60%	40%

Table 2 indicates that knowledge drivers are of greatest importance for the industry trying to develop their competitive advantage, with 71% of the answered classified as being of major importance. Thereafter, environmental drivers with 40% of the answers perceived to be of major importance and the equivalent percentage for technological drivers was only 17%.

Hence, it is an interesting finding that the industry considers technological drivers as being of least importance, yet the knowledge drivers are considered as much more important for the development of the industry. These drivers are connected, displaying two different sides of the same basic requirement, to develop the industry by either improving capabilities and production by advances in R & D or optimizing the output by improved market understanding. Thus, it could be indicative that Swedish companies producing wooden single-family houses have taken a low-cost development approach with a relatively conservative strategy enhancing their competitive advantage. This is emphasized by the importance put on the environmental drivers, which reflects both the strength of concrete and the lack of understanding of wood as a suitable building material, combined with their view that the industry imbalance requires change in regulation to further facilitate required development. One could argue that this is a relatively passive approach combined with how the industry value technological drivers compared to the other identified drivers.

More multi-family houses are planned to be built in wood, yet companies who can do that are limited in comparison to the demand. Further research should therefore focus on how the industry can increase their competitive position by enhancing the identified drivers. Thereby creating further understanding of how these drivers interact with the market requirement found in the procurement process. Studying this process would identify discrepancies between market expectations and industry

capabilities by increased end-to-end transparency throughout the building process, which can facilitate an increased rate of NPD based on the general market demand of an increased availability of housing units in Sweden.

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CRM (CUSTOMER RELATIONSHIP MANAGEMENT) IN SMEs IN WOOD INDUSTRY

Adina CANDREA
Transilvania University of Brasov, Faculty of Economic Sciences
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: adina.candrea@unitbv.ro

Cristina ANTONOAIE
DRESMARA
Str. Mihai Viteazul nr. 160, 500183 Brasov, Romania
E-mail: antonoaie.cristina@crmra.ro

Abstract

A scientific research was carried out using a 20 question form, addressed to the 30 managers from forestry and wood industry SMEs in Brasov. The form was filled out by 22 of them. We wanted to determine the awareness degree in regard to CRM (Customer Relationship Management). To analyse the data the SPSS software and Microsoft Office Excel were used. The research reached the conclusion that the majority of the managers of wood industry SMEs in Brasov are not ready and are not willing to invest in a CRM solution in the near future.

Key words: *CRM; vision; mission; information system; customer experience.*

INTRODUCTION

In today's dynamic economy companies need enabling technologies and tools in order to maximize the degree of satisfaction of their customers. CRM (Customer Relationship Management) represents a technology that can be implemented rapidly with relatively limited costs.

From the numerous definitions of CRM we selected two that we considered the most representatives for the objectives of this paper.

Investopedia defines CRM as follows: *"Customer relationship management (CRM) refers to the principles, practices and guidelines that an organization follows when interacting with its customers. From the organization's point of view, this entire relationship encompasses direct interactions with customers, such as sales and service-related processes, and forecasting and analysis of customer trends and behaviors. Ultimately, CRM serves to enhance the customer's overall experience."* (www.investopedia.com/terms/c/customer_relation_management.asp).

Gartner, Inc. (NYSE: IT) is the world's leading information technology research and advisory company. Gartner defines CRM as a "*business strategy with outcomes that optimize profitability, revenue and customer satisfaction, organized around customer segments, fostering customer-satisfying behaviors and implementing customer-centric processes."*

Also, Gartner created a CRM framework, or map, called "The Eight Building Blocks of CRM" (Fig. 1) based on an analysis of several hundred organizations that had successful CRM initiatives in 2002. The eight building blocks represent the main categories of topics for which Gartner clients have an interest in. The most successful organizations had a clear view of what they were doing in each of the eight areas, whereas the less successful had only a partial overview.

Fig. 1.
The Eight Building Blocks of CRM.
Source: Gartner (August 2016)

SAP considers that there are five main strategies that companies can employ to survive and thrive during uncertain economic conditions:

1. Focus on existing customers
2. Maximize revenue opportunities
3. Do more with less
4. Reduce operational costs
5. Optimize existing IT assets

OBJECTIVE

The main objective of the paper was to determine the awareness degree regarding CRM in wood industry SMEs in Brasov. The sample was formed of 30 companies and we obtained complete responses from 22 of the managers.

The general hypothesis from which we started was that the majority of the managers of the wood industry SMEs in Brasov are not currently using a CRM solution in their companies and are not willing to invest in such a solution in the near future.

MATERIAL, METHOD, EQUIPMENT

The activity fields of the 22 companies are forestry and other related activities, forestry felling activities and wood industry manufacturing industry. The denomination that we will use in this paper will be "wood industry SMEs", because only 5 companies out of 22 are active in the forestry field. 17 have an average number of employees between 10 – 49, 4 between 50 – 249 and 1 over 250 employees. 5 out of the 22 companies have been on the market for 5 – 10 years, 10 for 10 – 15 years and 7 for over 15 years. The representativeness of the sample is correct.

RESULTS AND DISCUSSION

The research was based on a number of 15 hypotheses.

Hypothesis no. 1: Usually wood industry SMEs do not have a Vision Statement.

Only 8 (36.4%) of the 22 managers that have answered to the question declared that their company has a Vision Statement. Hypothesis no. 1 was confirmed.

Hypothesis no.2: Usually wood industry SMEs do not have a Mission Statement.

This hypothesis was also confirmed, because the most common response was „No" – 12 out of 22 managers (54.5%).

Hypothesis no. 3: Managers of the wood industry SMEs are knowing the following notions: CRM (Customer Relationship Management), ICT (Information and Communication Technology), ERP (Enterprise Resources Planning), MRP (Material Requirements Planning), DSS (Decision Support System), ISS (Intelligent Support System), SCM (Supply Chain Management), SOA (Service Oriented Architecture).

As you can see in the table below, the most known notion was ICT (100% of responses were "well known"), followed by MRP (36.4% "known" and 63.6% "well known"), CRM (40.9% "known" and 59.1% "well known"), SCM (59.1% "known" and 40.9% "well known") and ERP (63.6% "known" and 36.4% "well known"). The notions of DSS and ISS were only somewhat known and medium known. So Hypothesis no. 3 was confirmed for 5 out of 7 notions.

Table 1

How well do you know the following terms?

Q3_known_item					
	Barely known	Somewhat known	Medium known	Known	Well known
Q3_known_item_CRM				40.9	59.1
Q3_known_item_ICT					100.0
Q3_Known_item_ERP				63.6	36.4
Q3_known_item_MRP				36.4	63.6
Q3_known_item_DSS		22.7	40.9	36.4	
Q3_known_item_ISS		45.5	31.8	22.7	
Q3_known_item_SCM				59.1	40.9

Hypothesis no. 4: wood industry SMEs are not usually using information solutions in their activity.

The most common answer to the question: "Does your company use any of the above mentioned information solutions?" was "No" (17 out of 22, meaning 77.3% of the total). Hypothesis no. 4 was confirmed.

Hypothesis no. 5: Managers of the wood industry SMEs had several sources of information about CRM (Customer Relationship Management).

The sources of information about CRM were, in order of the frequency: a training session (8 responses), a workshop/conference (6 responses), the Internet (3 responses), a professional association (2 responses), and media and other sources (1 response each). We mention that we didn't obtain any response for the variant "personal research/interest" and "from other managers" and we have 1 response "I can't remember". Hypothesis no. 5 was confirmed.

Table 2

How did you find out about CRM?

$Q5 Frequencies		Responses	
		N	Percent
source_CRM[a]	Q5_source_CRM_media	1	4.50%
	Q5_source_CRM_workshop	6	27.30%
	Q5_source_CRM_training	8	36.40%
	Q5_source_CRM_association	2	9.10%
	Q5_source_CRM_net	3	13.60%
	Q5_source_CRM_others	1	4.50%
	Q5_source_CRM_not_remember	1	4.50%
Total		22	100.00%

Hypothesis no. 6: Wood industry SMEs do not usually use a CRM solution.

At the question: "Has your company ever used a CRM (Customer Relationship Management) solution/system?" the dominant answer (18 out of 22) was "No". Hypothesis no. 6 was confirmed.

Hypothesis no. 7: There are several reasons for the wood industry SMEs for not using a CRM solution.

The main reasons for not using a CRM solution are, in order: lack of funds (12.7% of responses), it is not easily accessible (11.1%), lack of specialists (9.5%) and it consumes too much time (9.5%), lack of information (7.9%), uncertainty of positive results (7.9%) and no specialty technical support (7.9%), not relevant to our current activity (7.1%), inefficient communication (6.3%) and no concrete results / concrete application (6.3%).

They were also mentioned as reasons: the poor quality of the CRM solution (4.8%), the fact that the manager never thought about it until now (2.4%), it is not necessary (2.4%) and other reasons (1.6%). Hypothesis no. 7 was confirmed – there were 14 different reasons mentioned by the managers for not using a CRM solution in their company.

Table 3

What do you think are the reasons for not using a CRM solution in your company?

$Q7 Frequencies		Responses		Percent of Cases
		N	Percent	
Not_using_CRM[a]	Q7_no_CRM_lack_of_funds	16	12.70%	100.00%
	Q7_no_CRM_lack_specialists	12	9.50%	75.00%
	Q7_no_CRM_lack_info	10	7.90%	62.50%
	Q7_no_CRM_ineff_comm	8	6.30%	50.00%
	Q7_no_CRM_no_positive_results	10	7.90%	62.50%
	Q7_no_CRM_not_accessible	14	11.10%	87.50%
	Q7_no_CRM_not_relevant	9	7.10%	56.30%
	Q7_no_CRM_no_concrete_results	8	6.30%	50.00%
	Q7_no_CRM_too_much_time	12	9.50%	75.00%
	Q7_no_CRM_no_tech_support	10	7.90%	62.50%
	Q7_no_CRM_poor_quality	6	4.80%	37.50%
	Q7_no_CRM_not_thought	3	2.40%	18.80%
	Q7_no_CRM_not_necessary	3	2.40%	18.80%
	Q7_no_CRM_others	2	1.60%	12.50%
	Q7_no_CRM_no_response	3	2.40%	18.80%
Total		126	100.00%	787.50%

Hypothesis no. 8: The chances that a wood industry SME will use in the near future a CRM solution are very small.

At the question "What are the chances that your company will use a CRM solution in the near future?", more than half of the managers (11) from the total of 18 that declared that their company never used a CRM responded "very small chance", 5 of them responded "small chance" and 2 "even chance". So, this hypothesis was confirmed.

Table 4

What are the chances that your company will use a CRM solution in the near future?

Q8_CRM_future		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	very small chance	11	50	61.1	61.1
	small chance	5	22.7	27.8	88.9
	even chance	2	9.1	11.1	100
	Total	18	81.8	100	
Missing	System	4	18.2		
Total		22	100		

Hypothesis no. 9: The managers of the wood industry SMEs want to improve the current information system in their companies.

18 managers out of 22 responded that they would like to change/improve the current information system in their company. Hypothesis no. 9 was confirmed.

Hypothesis no. 10: wood industry SMEs rarely improve their IS (hardware and software).

54.5% of the managers responded that their companies rarely improve their Information System and 27.3% once a year.

Table 5

How often does your company improve its IS (hardware and software)?

Q10_IS_improve		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	rarely	12	54.5	54.5	54.5
	once a year	6	27.3	27.3	81.8
	once every 6 months	2	9.1	9.1	90.9
	once a month	1	4.5	4.5	95.5
	more often	1	4.5	4.5	100
	Total	22	100	100	

Also, as a control question directly related to the last question analyzed we asked the managers about the total amount allocated for the information system (% of total revenue). As you can see in the table below 6 of them responded that they do not allocate any money for the information system and 8 of them responded that they allocate between 0 – 2%. So, Hypothesis no. 10 was confirmed.

Table 6

Total amount allocated for the information system (% of total revenue) was:

Q20_IS		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	not allocated	6	27.3	27.3	27.3
	0 - 2%	8	36.4	36.4	63.6
	3 - 5%	3	13.6	13.6	77.3
	over 5%	5	22.7	22.7	100
	Total	22	100	100	

Hypothesis no. 11: The managers of the wood industry SMEs are not willing to invest much in a CRM solution.

At the question "How much funds would you spend on a CRM solution?" 8 managers (36.4%) responded that they are not willing to invest in a CRM solution and 12 of them (54.5%) mentioned that they would invest under 1% of the Total Revenue of their company. Only 2 (9.1%) responded that they would invest between 1% - 5% of the Total Revenue. We mention that no manager responded that he would invest over 5% of the Total Revenue. Hypothesis no. 11 was confirmed.

Table 7

How much funds would you spend on a CRM solution?

Q11_funds_CRM		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	under 1% of your Total Revenue	12	54.5	54.5	54.5
	1% - 5% of your Total Revenue	2	9.1	9.1	63.6
	we are not willing to invest in a CRM	8	36.4	36.4	100
	Total	22	100	100	

The control question here was about the amount of the total revenue during the last year (2015).

Table 8

Total revenue 2015

Q19_total_revenue		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	under 300000 euro	5	22.7	22.7	22.7
	300001 - 600000 euro	8	36.4	36.4	59.1
	600001 - 1000000 euro	4	18.2	18.2	77.3
	over 1000000 euro	5	22.7	22.7	100
	Total	22	100	100	

Hypothesis no. 12: A CRM solution for a wood industry SME should be focused mainly on customer experience.

The managers would prefer that a CRM solution to be focused on, in order of the frequency of responses: Customer Experience (18.9%) and also Technology (18.9%), Processes (15.1%), Information and Insight (13.2%), Strategy (11.3%), Vision (9.4%), Organizational Collaboration (7.5%) and Metrics (5.7%). Hypothesis no. 12 was confirmed.

Table 9

If you would ever consider using a CRM solution, what would you like it to be focused on?

$Q12 Frequencies		Responses		Percent of Cases
		N	Percent	
Focus_CRM[a]	Q12_CRM_focus_vision	5	9.40%	50.00%
	Q12_CRM_focus_strategy	6	11.30%	60.00%
	Q12_CRM_focus_customer_xp	10	18.90%	100.00%
	Q12_CRM_focus_org_coll	4	7.50%	40.00%
	Q12_CRM_focus_processes	8	15.10%	80.00%
	Q12_CRM_focus_info	7	13.20%	70.00%
	Q12_CRM_focus_technology	10	18.90%	100.00%
	Q12_CRM_focus_metrics	3	5.70%	30.00%
Total		53	100.00%	530.00%

Hypothesis no. 13: A CRM solution for a wood industry SME should be easy to use.

14 out of 22 managers considered that a CRM solution should be easy and very easy to use. So, the hypothesis was confirmed.

Table 10

How easy to use do you think the CRM solution should be?

Q13_CRM_easy		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	very hard to use / only for the IT specialists	3	13.6	13.6	13.6
	hard to use / only for the marketing specialists	3	13.6	13.6	27.3
	neither - nor / neutral	2	9.1	9.1	36.4
	easy to use	10	45.5	45.5	81.8
	very easy to use	4	18.2	18.2	100
	Total	22	100	100	

Hypothesis no. 14: In general, wood industry SMEs are not ready to try to implement a CRM solution.

The majority of the managers (16 out of 22) considered that in the near future there are small and very small chances to use a CRM solution. 8 of them considered that their company is not ready to implement such a solution and also 4 of them considered that this is not even necessary. Hypothesis no. 14 was confirmed.

Table 11

Do you consider that your company is ready to try to implement a CRM solution?

Q13_CRM_implement		Frequency	Percent	Valid Percent	Cumulative Percent
Valid	NO	8	36.4	36.4	36.4
	YES	6	27.3	27.3	63.6
	not necessary	4	18.2	18.2	81.8
	I don't know / no response	4	18.2	18.2	100
	Total	22	100	100	

Hypothesis no. 15: The wood industry SMEs are not up to date with the latest information technology releases.

16 out of 22 managers considered that their company is not even up to date with the latest IT releases. That is another reason why they could not and will not implement a CRM solution. Also this hypothesis was confirmed.

The control question here was: "Is your company constantly in touch with software suppliers for updates?" 14 out of 22 managers admitted that their companies are not even in a permanent contact with the software suppliers.

CONCLUSIONS

All the 15 hypothesis that this research was based on were confirmed as follows:

- Less than 40% of the 22 companies have a vision statement;
- Less than 55% of the 22 companies have a mission statement;
- Managers of the wood industry SMEs are knowing the main IT notions: CRM, ICT, ERP, MRP, DSS, ISS, SCM, SOA;
- More than 75% of the companies are not using any of the above mentioned IT solutions;
- Managers had 6 different sources of information about CRM;
- More than 80% of the companies never used a CRM solution;
- 15 different reasons were mentioned for not using a CRM solution;
- The chances that a company will use in the near future a CRM solution are very small;
- Even if these companies improve rarely their Information System, more than 80% of the managers mentioned that they would want to improve this system;
- The managers are not willing to invest much in a CRM solution – more than 30% would not invest at all;
- The ones that would invest in a CRM solution will prefer as focus customer experience and technology;
- The CRM solution should be easy to use for every employee;

- More than 50% of the managers recognized that their employees are not ready for the implementation of a CRM solution because their companies are not up to date with the latest information technology releases and they are not constantly in touch with software suppliers for updates.

A CRM solution is not easy to implement because it requires board-level vision of the managers and appropriate leadership. Also this process involves potentially difficult changes to processes, enterprise culture and the organization as a whole. The employees are of course the key factor and their training and willingness to be part of this implementation are very important.

According to Techopedia „the most critical purpose of CRM is to manage each instance of the company's customer interaction. CRM manages, stores and disseminates customer information with many built-in tools that can be applied to raw data pertaining to a customer or any given category of customer. For example, data may be analyzed to segregate customers according to demographic, occupation and age etc."

That is one of the reasons why we consider that a CRM solution will be very useful for the wood industry SMEs.

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SITUATION OF WOOD-BASED PANEL INDUSTRY IN TURKEY

Kadir DOĞAN
SFC Integrated Forestry Products Industry Trade Corporation, Turkey
Tel:0090 366 2141009, Fax:0090 366 214 6247, E-mail:kadir.dogan7137@gmail.com

M. Hakan AKYILDIZ
University of Kastamonu, Faculty of Forestry, Turkey
E-mail: mhakyildiz@kastamonu.edu.tr

Abstract

In this study, production capacities, export and import figures of industrial organizations of MDF, Particle Board, OSB, Laminated flooring, and Plywood operating in production activities in the wood-based panel industry in Turkey and with respect to this figures, consumption quantities and raw material needs for production between 2011 - 2015 have been viewed. Besides, contribution of board industry sector to the economy and employment and position of the Turkish board sector within European and world countries have been examined. Within the scope of the study, primarily, sector representatives of board industry in Turkey are listed. Potential of the market has been examined by stating the export and import figures by the board product range, location of the factories in which they operating in production, their production capacities and product range. Raw material inputs which are very important for board industry and effects of these inputs to the unit price have been viewed. Position of Fiberboard (MDF), PB, and Laminated flooring products from Turkish wood-based panel industry within the European and world countries has been examined. Accordingly, Turkey is in 1st place in Europe and 2nd place in the world in MDF production, 3rd place in Europe and 5th place in the world in Particle Board production, and 7th place in the world in sum of PB and OSB. In production of Laminated flooring, it is in the 2nd place in Europe and 3rd place in the world. Approximately 20% of the sales of Western European countries has been carried out in Turkey. Also, it is in the 35th place in production of plywood which is among the other wood-based panel industry products and 25th place in veneer in the world. Contribution of the sector to the economy and employment is great; however, the main problem of the sector is that the supply of wood which is the main raw material is not sufficient and it is expensive compared to the European and other countries.

Key words: *MDF (medium-density fiberboard); particle board (PB); laminated flooring; plywood; Turkey.*

INTRODUCTION

Wood-based panel industry is named as wooden and forestry products, is one of the leading 58 sectors of Turkey, is included in the manufacturing industry. This sector was established by public finance in 1950s and it has established its basic structuring on timber, parquet, woodwork, and board (plywood, particle and fiber board) industrial concept. It has been among the first examples of enterprises and establishments privatized since 2000. Many enterprises of the sector which has been subjected to restructuring of the private sector and technological advancement continue to carry out production in its activity area. The sectors of furniture and paper products using wood and wood-based raw materials have been taken out of this sector. Non-wood forest products which has contained raw materials of many sector for the recent years have been added to the sector of forestry and wooden products, increased the product range of that sector, and provided the opportunity for new markets in export (TOBB, Assembly sector report of Turkish forestry products 2015).

Industrial sector of forestry products has an important place in national and international trade due to production size it has and the foreign trade volume. Within the industrial sector of forestry products producing products of different specifications by processing round wood obtained from forests, group of wood-based panels stands out due to developing and changing production and economic conditions (Yıldırım et al. 2016). It increases the need to the wooden board products that solid materials are not sufficient in cases requiring use of wood on wide surfaces. Products such as particle board, fiberboard, particle board, plywood, and veneer meets many needs since they are more effortless and economic than solid products and thus, they has attracted attention in both Turkey and the world in recent years (Yıldırım et al. 2015).

The size of the sector which provides employment to directly to four hundred thousand and totally one million persons is around 12 billion USD. The target for 2023 is 25 billion USD. This sector

has two important sub-sectors. The first one is furniture and decoration sector and the second one is the sector of particle board and fiberboard and wooden products which meet the need of semi-product of the furniture sector. (OAIB sector report 2015). In our country, establishment making production with the investments having increased in the recent years and advanced technology in world standards has been established in particle board and fiberboard sector and a leading capacity and production technology in the world have been attained. According to the 2015 sector report of Central Anatolian Exporters' Associations (OAIB), Turkey is in the 1st place in Europe and 2nd place in the world in production of Fiberboard (MDF) production, 3rd place in Europe and 5th place in the world in production of Particle Board production, and 2nd place in Europe and 3rd place in the world in Laminated flooring production. Totally 25 different firms continue to make production in 34 different locations in board sector. All companies making production in Turkey have been gathered under the roof of Particle-Fiberboard Industrialists' Association. Also, 79 establishments have the production capacity of 558.264 m^3 in the plywood sector registered to the database of Turkish Union of Chambers and Commodity Exchanges (TOBB).

Forestlands of the world are approximately 4 billion hectares (ha) and it is one-third of the total lands. Nearly all the forests consist of natural forests (95%) and a very little part of it does so of plantation forests (5%). Rates of continents to forests are 46% for Europe, 25.7% for North and Central America, and 21.8% for Africa. Russian Federation, Brazil, Canada, USA, and China which are the five richest countries in terms of forests have the half of total forestlands. (TOBB, Assembly sector report of Turkish forestry products 2015). In Turkey, according to the data obtained as the result of update on the database of forest management plans (ENVANİS) renewed between the years of 2013 and 2015, forestland of the country has been detected to be 22.3 million hectares. That forestland is 28.6% of the general area of the country. Wood assets increased to 1.6 billion m^3 in 2015. Forestland of Turkey covers 28.6% of the area of 78 million hectares. Forestlands without trees have not been added to these areas (OGM 2017). According to the information in forest management plans renewed today, annual average revenue obtained from forestlands has been determined to be as follows: From high forests: 15.942.459 m^3, From coppice forests: 2.372.162 m^3, Totally: 18.314.621 m^3. According to the information MDF and Particle Board Industrialists' Association, supply sources for wooden raw material required for MDF and PB: 1. From General Directorate of Forestry; fiber particle wood, wood for paper coming to the sector, firewood coming to the sector, 2. From private sector; poplar, willow etc., industrial wastes such as wood dust coming to the sector, 3. Imported from abroad; wood particle (chips) and woods.

In this compilation, production, export, and import figures of MDF, PB, OSB, Laminated flooring, plywood and board consumption quantities from wood-based panel industry in Turkey, Europe, and the world have been shown. Thus, it is tried to examine the change between production and consumption and to analyze the status of the board industry in next years. With the contribution of board industry to the Turkish economy and employment and its position in Europe and the world, importance of the board industry within the sector of forestry products is tried to be shown. With this presentation, it is targeted to strengthen the position of the Turkish panel industry having an important place in Europe and the world and improve the competition conditions with the other board industry establishments in Europe and the world.

MATERIAL AND METHOD

In this study, production, export, and import data of MDF - HDF, Particle Board, OSB, and plywood boards among the wood-based panel products in Turkey, Europe, and the world have been tried to be examined from FAOSTAT database. Production and sales data of laminated flooring by years have been taken from annual data of Domotex conference published on the website of EFPL, European laminated flooring parquet producers' establishment. Turkish board industry raw material need has been tried to be assessed with the data obtained from the report of MDF and Particle Board Industrialists' Association. Production and foreign trade data of these products have been compared to the European and world foreign trade data of the sector, position of the sector in Europe and the world has been probed, and up-to-date status of the sector has been interpreted with the help of numerical datas. Data of foreign trade of Turkey has been obtained from the database of Central Anatolian Exporters' Associations, TOBB Assembly sector report (2015), and TÜİK (Turkish Statistics Institution) (2015) database.

Definitions, characteristics, and area of use of the products subjected to examination have been stated below. These products, respectively, are;

Particle Board; product definitions and characteristics; Particle Board; "they are the boards obtained by gluing and laying of particles obtained from wood pieces (wood particles, saw dust, shavings etc.) and/or other lignocellulosic materials (from lignified plants such as flax, hemp harl,

water-extracted sugar cane pith, etc.) and mixing with hardening materials and materials providing the hydrophobical property, and pressing under temperature and pressure." 3-layers board production is realized in single or multiple-floor presses with or without interruptions in particle board sector (OAIB sector report 2015).

OSB board definition: According to EN 13986:2004, OSB is a multilayered boards consisting of wood particles (strands) in form of stripes longer than 50 mm and narrower than 2 mm with an adhesive. Particles in form of stripes on the outer layer have been aligned in parallel with the width and length of the board. Particles in the inner layer are randomly oriented or aligned and generally they are perpendicular to the particles in form of stripes on the outer surface. In the following Table 1, firm names, locations, establishments capacities, and active years of the industrial establishments of Particle Board and OSB operating in Turkey have been listed.

Table 1

PB and OSB Factories operating in Turkey and Installed Capacities

Nr.	Company	Place	Product	Capacity m³/day	Capacity m³/year	Year of operating
1	KASTAMONU ENTEGRE	BALIKESIR	PB	1.700	544.000	2005
		Gebze/KOCAELI		1.500	480.000	2010
		KASTAMONU		670	214.400	1975
		Terme / SAMSUN		550	176.000	1990
		Tarsus / MERSİN		500	160.000	1997
2	YILDIZ ENTEGRE	Mudurnu / BOLU		1.150	368.000	2012
		Akhisar / MANİSA		1.700	544.000	2012
3	STARWOOD	İnegöl / BURSA		150	48.000	1997
				950	304.000	1995
				1.750	560.000	2004
4	YILDIZ SUNTA MDF	İzmit / KOCAELI		1.700	544.000	2010
5	ORMA	ISPARTA		1.500	480.000	2012
				300	96.000	1978
				300	96.000	1981
6	TEVERPAN	Çerkezköy / TEKIRDAĞ		450	144.000	2001
7	SUNTASAN / KÜPELİLER	ESKIŞEHIR		300	96.000	1981
8	S.F.C. KRONOSPAN	KASTAMONU		200	64.000	1978
9	GENTAŞ A.Ş	BOLU		96	30.720	1992
10	DASAŞ ENTEGRE AĞAÇ	Devrek / ZONGULDAK		340	108.800	1975
11	VEZİRAĞAÇ	Vezirköprü / SAMSUN		175	56.000	1996
12	KÜPELİLER SİMAV	Simav / KÜTAHYA	OSB	300	96.000	1982
13	S.F.C. KRONOSPAN	KASTAMONU		300	96.000	2011
14	SUMAŞ A.Ş.	Edremit/ BALIKESİR		150	48.000	2004
PB and OSB TOTAL INSTALLED CAPACITIES				**16.731**	**5.353.920**	

According to the data of MDF and Particle Board Industrialists' Association, Particle Board and OSB factories operating in Turkey is 14 by the number of firms and 19 by the provinces. Installed capacity is daily 16.731 m³/day and annually 5,353,920 m³/year. Particle Board and fiberboard industries are within intensive relationship with furniture, forestry, glue and chemical material production sector, timber factories and carpenters, paper sector, construction sector (prefabricated house construction), decoration, wood traders, petroleum product sellers, automotive sector, energy sector, profile producers, forest-village cooperatives, cement production sector, and metal industry (OAIB sector report 2015). According to market researches, it is stated that purchase in the highest quantity in European particle board sector is made by furniture sector, 82% of particle board is consumed by furniture sector, and it is followed by construction, door, and other sectors (Turkish forest products sector assembly 2015). Particle Board and fiberboard industries are within intensive relationship with furniture, forestry, glue and chemical material production sector, timber factories and carpenters, paper sector, construction sector (prefabricated house construction), decoration, wood

traders, petroleum product sellers, automotive sector, energy sector, profile producers, forest-village cooperatives, cement production sector, and metal industry (OAIB sector report 2015). OSB activities in Turkey continue in three provinces in daily 750 m³/day and annually 240,000 m³. By FAOSTAT 2016 data, PB and OSB production quantities in Turkey, Europe, and the world in the last five years between 2011 and 2015.

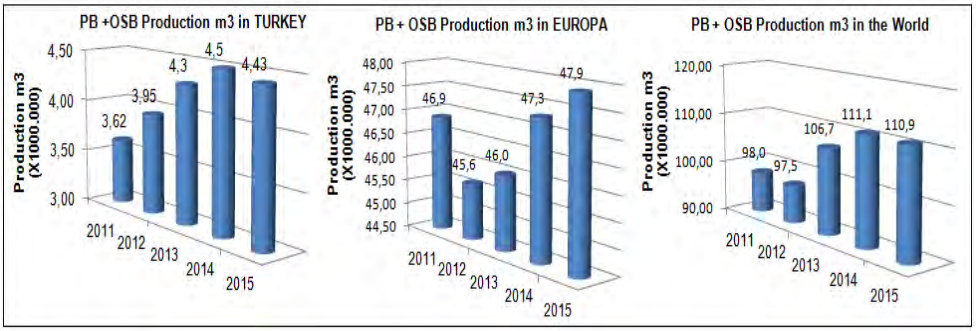
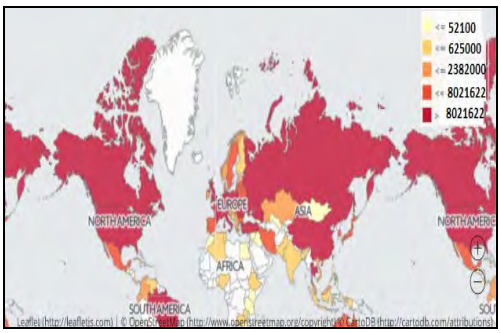


Fig. 1.
PB and OSB Production Quantities

PB and OSB production map in the world is shown in figure 2 and graphic of Particle Board and OSB distribution rate is shown in figure 3 (FAOSTAT 2016).



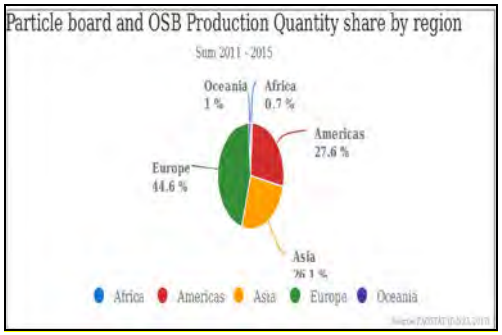


Fig. 2.
PB and OSB production map

Fig. 3.
Particle Board and OSB distribution rates

From 2011 to 2015, total production quantity of PB and OSB in Turkey, Europe, and the world has respectively increased approximately by 22%, 2,2%, and 13%. The biggest increase in total production quantity of PB and OSB is observed in Turkey. Particle Board and OSB production distribution rates regionally based on FAOSTAT data are shown in figure 3. PB and OSB consumption in Turkey have increased until 2013; however it has decreased by 0.96% between 2014 and 2015. Change in consumption is shown in Table 2.

Table 2

Turkey PB + OSB Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	3.62	3.95	4.30	4.50	4.43	-1.56
Import	0.33	0.48	0.43	0.27	0.22	-15.85
Export	0.32	0.32	0,31	0.48	0.41	-14.76
Apparent Consumption	3.64	4.11	4.42	4.28	4.24	-0.96

PB and OSB consumption in Europe have decreased until 2013; however it has increased by 0.73% between 2014 and 2015. Change in consumption is shown in Table 3.

Table 3

European PB + OSB Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	46.9	45.6	46,0	47.3	47.9	1.26
Import	13.08	13.17	13.95	14.99	15.15	1.08
Export	15.48	16.76	17.04	18.04	18.47	2.41
Apparent Consumption	44.48	42.06	42.93	44.25	44.57	0.73

PB and OSB consumption in the world have constantly increased until 2015; however it has changed much with the rate 0.03% between 2014 and 2015. Change in consumption is shown in Table 4.

Table 4

World PB + OSB Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	98.0	97.5	106.7	111.1	110.9	-0.12
Import	23.45	23.75	25.73	27.31	27.96	2.38
Export	23.20	25.02	25.94	28.05	28.54	1.75
Apparent Consumption	98.29	96.22	106.53	110.33	110.35	0.03

According to the annual assessment data for forest products in 2015; apparent consumption of particle board has decreased by 0.6% in Europe. The highest five consumer market for particle board (respectively; from top to down); Germany, Poland, Turkey, Italy and England. Total consumption is equal to 60% of Europe. In 2015, area of use of particle board is generally is furniture industry and other application areas are in construction sector in Europe. OSB consumption has been 5.5 million m³ in Europe and the rate has increased by 5.5% (Forest Product Annual Review 2015).

Fiberboard (MDF) definition: It is defined as "the boards produced as the result of forming by using special adhesive material and benefiting from natural adhesion and felting characteristics of vegetable fiber and fiber clusters". MDF board industry has first been established in Ordu in 1985 in Turkey. Fiberboard sector is included in Article "44" with the 12 digits "Fiberboards from wood and other non-wooden materials (whether agglomerated with resins or other organic materials" with the GTSP number "4411". Particle Board and fiberboard industries are within intensive relationship with furniture, forestry, glue and chemical material production sector, timber factories and carpenters, paper sector, construction sector (prefabricated house construction), decoration, wood traders, petroleum product sellers, automotive sector, energy sector, profile producers, forest-village cooperatives, cement production sector, and metal industry (OAIB sector report 2015).

According to the data of MDF and Particle Board Industrialists' Association, MDF factories operating in Turkey is 15 by the number of firms and 20 by the provinces. Installed capacity is daily 21.185 m³/day and annually 6,779,200 m³/year. Some firms have multiple MDF factories in the same province. Fiberboard (MDF and HDF) is used in raw form and in a decorative melamine-surfaced form intensively in furniture sector since it is easy to process and its resistance and quality values are high. Also, it is intensively used in door sector as well as it is easily preferred in construction sector, decoration, and various applications. Between the years of 2011 and 2015, MDF and HDF production quantities in Turkey, Europe, and the world are given in Figure 4.

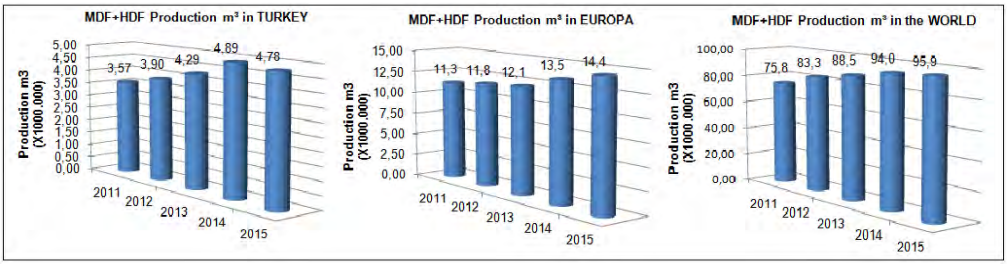


Fig. 4.
MDF and HDF Production Quantities.

From 2011 to 2015, total production quantity of MDF and HDF in Turkey, Europe, and the world has respectively increased approximately by 34%, 28%, and 26%. The biggest increase in total production quantity of MDF and HDF is observed in Turkey. MDF factories operating in Turkey are shown Table 5.

Table 5

MDF Factories operating in Turkey and Installed Capacities

Nr.	Company	Place	Product	Capacity m³/day	Capacity m³/year	Operating year
1	YILDIZ ENTEGRE A.Ş	İzmit / KOCAELİ	MDF	1.200	384.000	2007
				1.200	384.000	2006
				1.200	384.000	2001
				1.200	56.000	2009
		Tarsus / MERSİN		1.325	424.000	2012
2	KASTAMONU ENTEGRE	Gebze / KOCAELİ		770	246.400	1994-2009
				580	185.600	2003-2012
		KASTAMONU		1.200	384.000	2008
		ADANA		1.325	424.000	2011
3	YILDIZ SUNTA MDF A.Ş	İzmit / Kocaeli		600	192.000	1997
				1.200	384.000	2009
4	A.G.T A.Ş	ANTALYA		1.325	424.000	2013
5	TEVERPAN MDF A.Ş	Çerkezköy / TEKİRDAĞ		280	89.600	1999
				280	89.600	2001
		Veliköy / TEKİRDAĞ		600	192.000	2005
				175	56.000	2009
6	DİVAPAN A.Ş	DÜZCE		750	240.000	Assembly
				320	102.400	1999
7	ÇAMSAN POYRAZ A.Ş	ORDU		560	179.200	1995
				260	83.200	1985
8	STARWOOD A.Ş	İnegöl / BURSA		600	192.000	2009
				1.325	424.000	2016
9	ÇAMSAN ENTEGRE A.Ş	Hendek / ADAPAZARI		700	224.000	2004
		ADAPAZARI		1.300	416.000	2016
10	VEZİRAĞAÇ A.Ş	Vezirköprü / SAMSUN		600	192.000	2008
11	S.F.C A.Ş KRONOSPAN	KASTAMONU		475	152.000	2003
				900	300.000	Assembly
12	SELOLİT A.Ş	MANİSA		60	19.200	1976
13	BALKANLAR MDF A.Ş	KIRKLARELİ		200	64.000	2013
14	S.B.S A.Ş	M.Kemalpaşa / BURSA		300	96.000	2009
15	BEYPAN (MEHTAP) A.Ş	KAYSERİ		300	96.000	2015
MDF TOTAL INSTALLED CAPACITIES				**21.185**	**6.779.200**	

MDF and HDF production map in Europe and the world is shown in Figure 5 and world MDF and HDF distribution rates are shown in Figure 6 (FAOSTAT 2016).

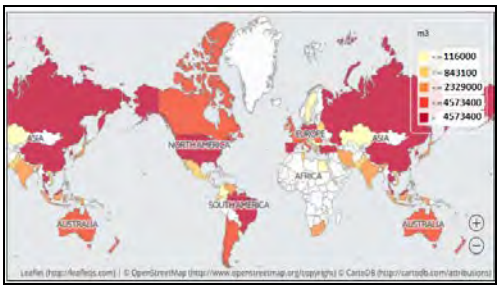


Fig. 5.
World MDF and HDF production map.

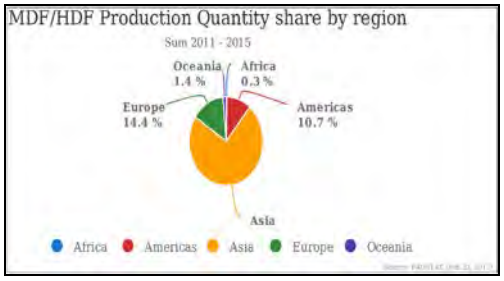


Fig. 6.
World MDF and HDF regional distribution.

Turkey is in the 2nd place after China in production of MDF and HDF board production. In Figure 7, the top 10 countries in production of MDF and HDF production are shown. Among the European countries, Poland is in 5th place and Germany is in the 9th place in world ranking.

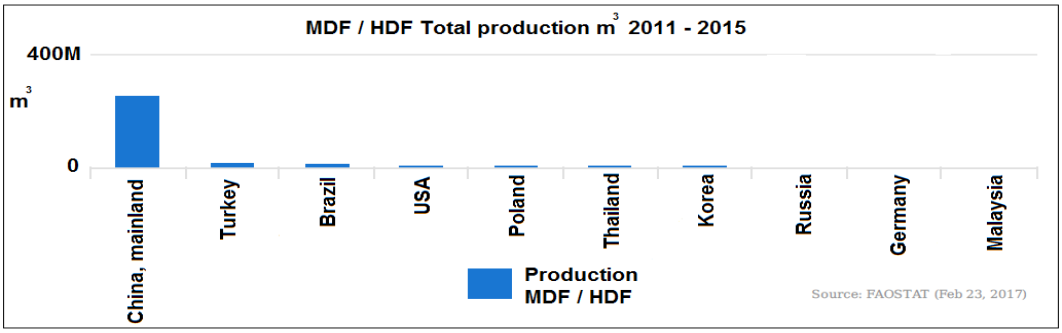


Fig. 7.
Top 10 in World MDF and HDF production.

In Turkey, MDF and HDF board consumption has significantly increased between 2011 and 2014 and decreased by 4.12% in 2015. Change in consumption figures is shown in Table 6.

Table 6

Turkey MDF + HDF Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	3.57	3.90	4.29	4.89	4.78	-2.21
Import	0,31	0.42	0.33	0.25	0.22	-13.39
Export	0.56	0.47	0.36	0.46	0.51	11.14
Apparent Consumption	3.33	3.86	4.26	4.68	4.49	-4.12

No considerable change has occurred in consumption figures between 2011 and 2015 in Europe. An increase by 0.73% is seen in 2015 compared to 2014. Change in consumption figures is shown in Table 7.

Table 7

European MDF + HDF Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	46.9	45.6	46,0	47.3	47.9	1.26
Import	13.08	13.17	13.95	14.99	15.15	1.08
Export	15.48	16.76	17.04	18.04	18.47	2.41
Apparent Consumption	44.48	42.06	42.93	44.25	44.57	0.73

MDF and HDF consumption in the world has continuously increased until 2015 and consumption has increased by 2.61% in 2015 compared to 2014. Change in consumption is shown in Table 8.

Table 8

World MDF + HDF Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	75.8	83.3	88.5	94.0	95.9	2.09
Import	14.42	15.00	14.72	15.04	15.76	4.74
Export	15.98	15.96	16.21	16.93	17.21	1.64
Apparent Consumption	74.27	82.39	86.98	92.07	94.47	2.61

According to the annual assessment data for forest products in 2015; consumption of MDF has decreased by 2.6% in Europe in 2015. Main users of MDF board have been laminated flooring (34%) and furniture sector (45%) among the sub-sectors.

İbrahim Yıldırım et al. (2015) has made their future estimations in relation with consumption quantities from 1995 to 2013 and consumption quantities to 2020 by using multiple regression method in the study which they conducted with the name of projection and economic status of wood-based board sector in Turkey. Accordingly, it is estimated that MDF consumption quantity in Turkey in 2020: 5.3 million m³, PB consumption quantity: 7 million m³, 0,7 million m³.

According to the report of MDF and PB Industrialists' Association, board sector ranking in Europe is shown in Table 9.

Table 9

European board industry ranking

MDF m³ / year			**PB m³ / year**			**Laminate flooring m² / year**		
1	**TÜRKİYE**	**5,54**	1	RUSYA	6,64	1	ALMANYA	272
2	ALMANYA	3,79	2	ALMANYA	5,52	**2**	**TÜRKİYE**	**110**
			3	**TÜRKİYE**	**5,30**	3	RUSYA	75

Laminated flooring: It is a flooring board consisting of special protector and transparent film layer on the top, wood textured decorative paper under it, E1-quality HDF, MDF, particle board etc. board, and finishing containing melamine increasing strength and protecting from moisture (TOBB assembly sector report 2015).

Laminated flooring - product structure

(1) Upper layer of melamine coating which is highly resistant to wearing (Overlay)
(2) Decor layer on which decors are printed (Decor paper)
(3) Bearing board from MDF or HDF
(4) Backside (Balance paper)
(5) Easy-to-floor by its interlocking mechanics and easy-to-repair by its low structural height (EPFL, 2016)

Laminated flooring firms launch their products under various brands and different names under these brands. Important laminated flooring firms operating in Turkey and their known brands are shown in Table 10 and firms operating in Europe and member to European Laminated Flooring Producers' Association (EPFL) and their countries are shown in Table 11.

Table 10

Laminated Flooring Firms Operating in Turkey and Their Brands

Company	Çamsan	Kastamonu Integrated		Yıldız Integrated	Vezir Köprü Wood Products	AGT	Yıldız Sunta MDF	Işık Ahşap	SFC Integrated Wood Product
Brand	ParkeLam	Floorpan	Artfloor	Vario Click	Peli Parquet	AGT Parquet	Moonlock	Işık Parquet	Dafne Parquet

Table 11

Some firms operating in Europe and member to European Laminated Flooring Producers' Association (EPFL)

Company	Akzenta	Alsapan Flooring	Baterio Laminate	Egger	Kindl	Krono	Swiss	Skema	Pergo
Country	Germany	France	Belgium	Germany	Austria	Germany	Switzerland	Italy	Belgium

According to 2016 statistical data compiled and published from the reports given by EPLF member countries, total sales of laminated flooring in 2016 have been 476 million m². 2007-2016 laminated flooring sales statistics are given in Figure 8.

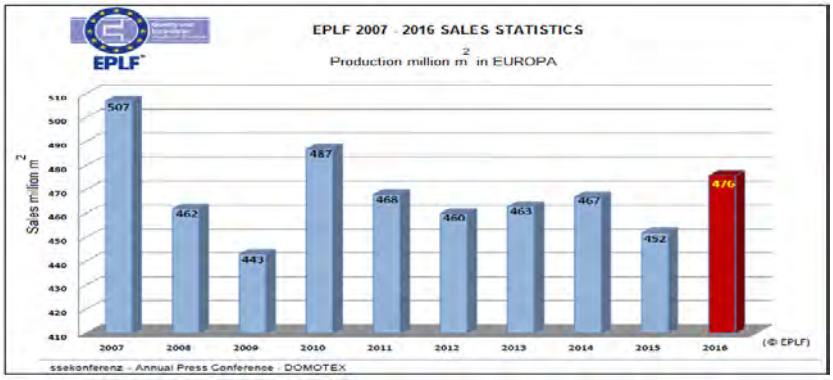
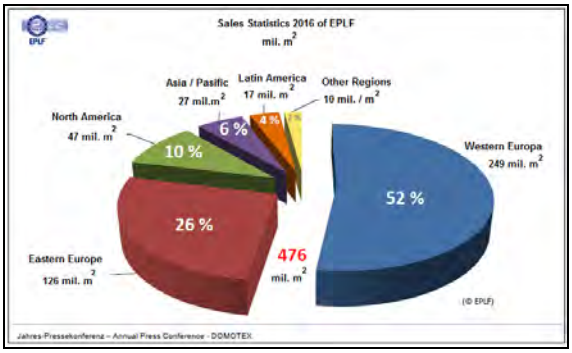


Fig. 8.
Laminated flooring sales statistics.

2016 laminated flooring sales rates of EPLF member countries are shown in Figure 9. Turkey has been included into Western Europe region. 2016 laminated flooring sales rates of EPLF member countries from Western Europe are shown in Figure 10.



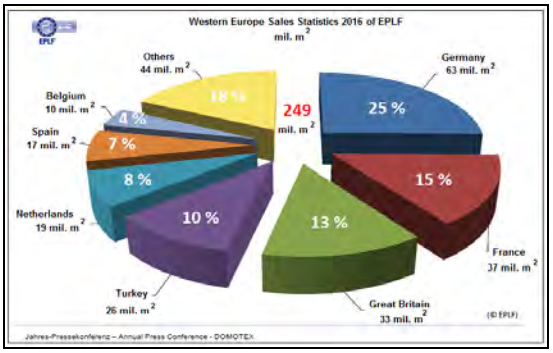


Fig. 9.
Laminated flooring sales rates.

Fig. 10.
Western European countries flooring sales rates.

Plywood's definition is that they are the panels formed by arranging and adhering wooden veneers in a way which fiber directions shall be perpendicular to each other. Plywood consists of minimum three layers. 74 establishments have the production capacity of 479.582 m³ in the plywood sector registered to the database of TOBB. Provinces in which the establishments intensify are Adapazarı, Kastamonu, Bolu, İstanbul, Ankara, Bursa, and Çorum. Assembly sector report of Turkish forest products (TOBB 2015). According to sector report of İzmir Chamber of Commerce (İZTO 2014), Turkey is in 35th place among 126 countries in production of plywood with 200 thousand m³ production and 0.1% share. Plywood is a product having a wide range using advantage such as construction sector, packaging industry, advertisement stands, and traffic signs, Central Anatolian Exporters' Associations Plywood report (OIAB 2011). There are many producers and export-import firms operating in plywood sector. Important establishments among them are shown in Table 12.

Table 12

Some Plywood Industry Firms Operating in Turkey

Company	Bizon Wood Industry	Dastaş Demirci	Hasep Kaplama	Taner Integrated Wood Product	Yiğit Construction	Sitaş Siteliler	Ekol Plywood	TKS	Çağ Plywood	Starwood Forest	Bahar Forest
City	Adapazarı	Adapazarı	Düzce/Bolu	Bursa	İstanbul	Ankara	Kastamonu	Kastamonu	Kastamonu	İnegöl/Bursa	İstanbul

Plywood production quantities in Turkey, Europe, and the world are shown in Figure 11 (FAOSTAT 2016).

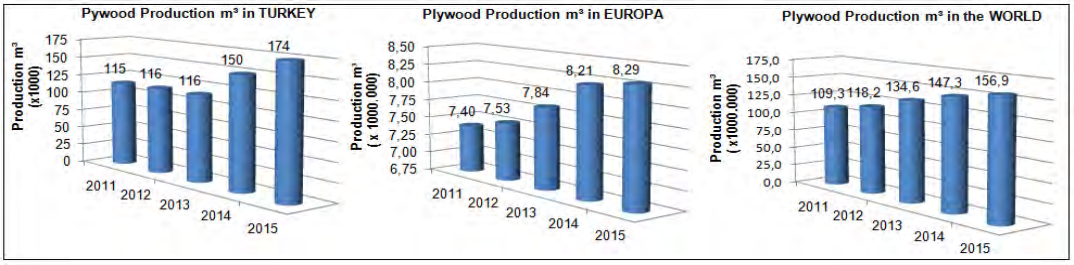


Fig. 11.
Plywood production quantity.

Plywood production has increased by 51.3% in Turkey, 12.0% in Europe, and 43.4% in the world in 2015 compared to 2011. In 2015, production in Turkey has been 2.1% of production in Europe and 0.1% of total Plywood production in the world. World plywood production map is shown in Figure 12 and Plywood production regional distribution is shown in Figure 13.

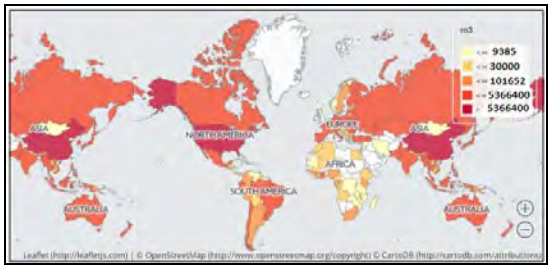


Fig. 12.
Plywood production map

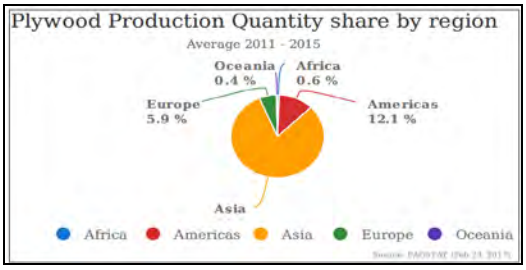


Fig. 13.
World plywood production regional distribution.

When distribution of plywood production in the world is examined, it is seen that the biggest production is made in Asian countries and the lowest production is made in Oceania and African countries. The top 10 countries in which the plywood production is the highest are shown in Figure 14.

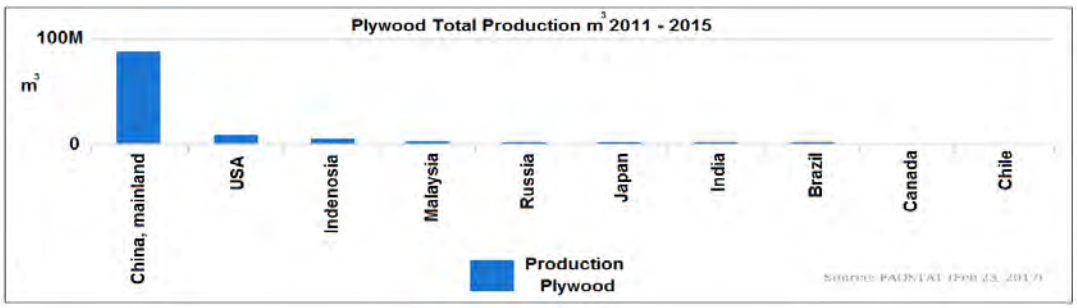


Fig. 14.
The top 10 countries in which the plywood production is the highest.

While China is in the 1st place in plywood production in the world, the second place belongs to United States of America. Plywood consumption in Turkey has increased until 2015 and no change is observed in 2015 compared to 2014. Annual change in consumption is shown in Table 13.

Table 13

Turkish Plywood Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	115	116	116	150	174	16,0
Import	244	268	293	293	279	-4.8
Export	17.5	17.3	4.4	4.2	14.0	237.1
Apparent Consumption	341.5	366.7	404.6	438.8	439.0	0.0

Between 2011 – 2015, plywood consumption in Europe has not changed. It has decreased 1.5% between 2014 and 2015. Annual changes in consumption are shown in Table 14.

Table 14

European Plywood Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	7.40	7.53	7.84	8.21	8.29	1.0
Import	6.86	6.63	6.57	7,14	7.23	1,2
Export	5.51	5.52	5.81	6.25	6.56	5.0
Apparent Consumption	8.7	8.6	8.6	9.1	9.0	-1.5

Between 2011 – 2015, plywood consumption in the world has increased. It has increased by 1.5% between 2014 and 2015. Annual changes in consumption are shown in Table 15.

Table 15

World Plywood Consumption (million m³)

	2011	2012	2013	2014	2015	Change % 2015-2014
Production	109.32	118.18	134.62	147.34	156.85	6.5
Import	23.47	24.05	23.55	24.51	25.09	2.3
Export	25.32	25.85	26.49	28.11	28.62	1.8
Apparent Consumption	107.5	116.4	131.7	143.7	153.3	6.7

Turkish Board export; according to the TÜİK (Turkish Statistics Institution) data published in sector report of Central Anatolian Exporters' Associations, it is seen that while export to Iran in 2010 was around 154.1 million $, it decreased to 101.8 million $ in 2013, this value increased to 131.9 million $ in 2014 and Iran has become in the 1st place. Other markets showing continuous increase in export from 2010 to 2014 are Turkmenistan, Algeria, Romania, and England. 2nd biggest market of Turkey in board export is Iraq. Export of totally 52.5 million $ was made to Iraq in 2014. Export value of Georgia which is the 3rd biggest market was recorded as 47.4 million $ in 2014. While export made to Azerbaijan which is in the 4th place was 19.6 million $ in 2010, this value has increased to 38.2 million $ in 2014. While export made to Algeria which is among the other markets of which export volume always increase was 2.6 million $ in 2010, this value has increased to 8.2 million $ in 2014. In the period from 2010 to 2014, our exports to Romania, England, and Lebanon have respectively increased by 221.4%, 455%, and 408%. According to (FAOSTAT 2016) data, change in export quantities in PB, OSB, MDF-HDF, and Plywood in Turkey between 2011 and 2015 is shown in Figure 15.

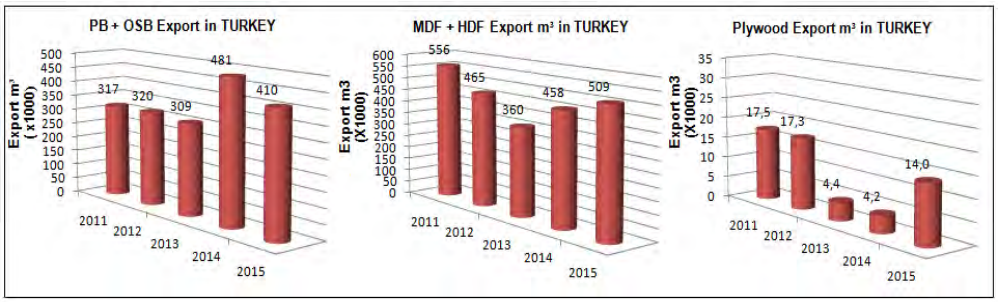


Fig. 15.
Change in export quantities in Turkey.

According to these data, while the highest export volume was in MDF-HDF, the lowest one is in Plywood. It is understood from the graphics that a decrease of 14.7% has occurred in Particle Board and OSB board export and increase of 11.1% has occurred in MDF and HDF export in Turkey in 2015 compared to 2014. It is seen for plywood export that very low rate of export has occurred between 2013 and 2014; however it increased again in 2015. According to TradeMap data of 2013, of world board export, 20.3% is realized by People's Republic of China, and then by Germany, Indonesia, and Malaysia respectively by 9.1%, 6.8%, and 6.5%. Turkey is ranked at the 21st place among 139 exporters (OAIB sector report 2015).

Board import: According to TÜİK (Turkish Statistics Institution), when board import is examined based on products, it attracts attention that Turkey made highest amount of import in plywood with 335 million $ and it is followed by MDF and fiberboard import with 223 million $. Particle board and OSB import is in the 3rd place with 78.5 million $. When the most important markets in board import of Turkey are examined, while Romania is in the 1st place with 128.7 million $, it is followed by Russian Federation with 125.7 million $, and Germany is in 3rd place with 76.5 million $ (OAIB sector report 2015). When import quantities are examined, a decrease is seen in PB and OSB productions, MDF-HDF production, and Plywood production until 2015. According to Trademap data of 2013, 14,3% of world board import is made by USA. It is followed by Japan (8.6%), Germany (6.6%), England (4.1%), and France (3.5%). Turkey takes place on the 12th place in board importing of the world among 225 board importers (OAIB sector report 2015). Change in import quantities in PB, OSB, MDF-HDF, and Plywood in Turkey between 2011 and 2015 is shown in Figure 16 (FAOSTAT 2016).

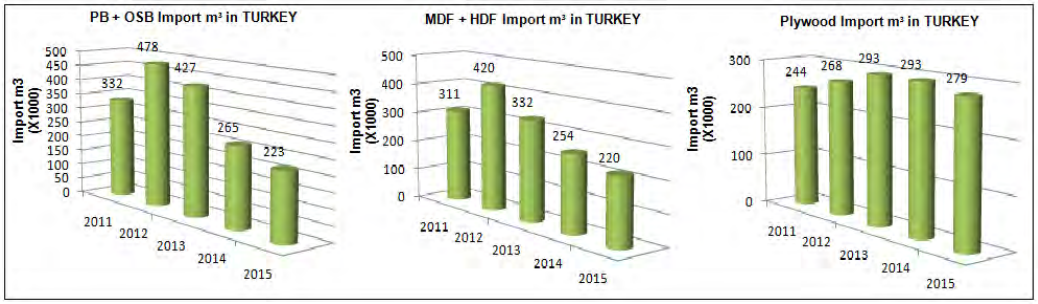


Fig. 16.
Change in import quantities in Turkey.

It is seen from the graphics that imports of Particle Board and OSB imports, MDF and HDF board imports, and Plywood imports respectively in Turkey in 2015 respectively by 15.8%, 13.4%, and 4.8% compared to 2014. It is seen that the highest import quantity has occurred in Plywood industry.

The sector of wooden products and forestry products contains production and trade of many goods out of wood-based boards, particle board, OSB, and fiberboard. Primary items among them are log and timber, solid products made of timber (parquet etc.), finger joint-added solid profile, laminated wooden boards (plywood, veneer), and packaging. Installed capacity using rate of board industry varies between 75% and 80% (OIAB sector report 2015). Chips and wood import is made from abroad since wood and wood-based raw material needed by the sector and corresponding to the capacity using rate of the sector cannot be met from wood and industrial wood waste provided by General Directorate of Forestry and private sector. The main problem of the board industry is that the supply of

wood which is the main raw material is not sufficient and it is expensive compared to the international markets. Wood prices in Turkey are two times expensive compared to Europe and three times expensive compared to United States of America, Brazil, Canada, Venezuela, Ukraine, and Russia. The share of the wooden raw materials in the board cost is around 45-53%. Turkey uses the most expensive wood of the world and 30-35% of the total wooden raw material need is met by import wood and wood particles (chips). Although the supply quantity of the wood needed by the sector from internal sources as the result of the studies made by General Directorate of Forestry has increased to 16 million m³ in 2013 from 7 million m³ in 2002, it is not sufficient. For all the needs of the sector to be able to be met from the internal sources, studies of General Directorate of Forestry continue. Maintenance of the forests and sales of General Directorate of Forestry are of great importance for sub-industrial board sector using industrial wood which is thin and not very valuable. Thus, it is required that wooden raw material production in Turkey is to be increased in a way which shall meet the needs of the sector and to be brought to the price level which shall allow international competition. 2015 production capacity of the sector is 9,000,000 - -10.000.000 m³/year. For this production, approximately 22 - 24 million stere/year wooden raw material is needed. 25-30% of total raw material need of our sector is supplied from abroad and 70-75% of the same is done so from domestic market.

CONCLUSION

In parallel with the demand to the board industry products in Turkey, increase is apparently seen in number of factories and production capacities. It is observed that there has been increase in Turkey, Europe, and the world in PB and OSB production between the years of 2011 and 2015; however, the biggest increase in the same has occurred in Turkey with the rate of 22%. In PB production, while Turkey is in 3rd place in Europe and 5th place in the world, it is in the 7th place in sum of PB and OSB in the world after Poland. That is because Turkey has fallen behind in OSB production. While there is increase of 29% in export of PB and OSB, a decrease of 32% is seen in import. Contribution of PB and OSB board industry to economy and its contribution to employment with the increasing production capacity and number of facilities are seen from these figures. Increase of 34% has occurred in total production of MDF and HDF in Turkey. While 8.5% decrease is seen in export, 29% decrease has occurred in import. Production capacity of MDF and HDF board industry meets the needs of Turkish market. In parallel with the development of Turkish board industry in the recent years, position it has attained in Europe and the world is so big and important that it cannot be ignored. It asserts the technology, quality and the importance of the sector in the country that Turkey is the 21st biggest board exporter among 139 exporter countries. With the export figures and employment of one million people, the contribution of the sector to the economy is considerable. Although forestland rates and industrial wood sources of Turkey has increased, board industry has had to import 30-35% of total wooden raw material need as wood and wooden particles from abroad since it cannot meet the increasing capacity needs of it. Insufficient wood sources as well as very high cost of wood due to various reasons stand as the biggest barrier competition and development of board industry of which main cost element is wood. The main inputs in the sector are wood and glue and other additives are fuel and energy. Proportionately; the costs are as follows: wood is 42-53%, glue 21-24%, energy is 11%, operation, maintenance, labor, general management, and sales and marketing, and depreciation are 20% (OIAB sector report 2015). As seen from the figures, wooden raw material is in the first place in board costs and it is as much as the sum of other costs. Thus, supply of wooden raw material in the board industry is very important in terms of board costs due to high level of wood prices as well.

Consequently, it is required that the purchasing costs of wood and log are to be decreased for the sector of wood and forest products to have a voice and to be able to compete in international markets. High level of added value obtained from board industry products is important for turkey due to its contribution to the economy and employment. It may be recommended that emphasize is to be laid on production technologies with alternative raw materials due to raw material problem likely to be experienced in the future. Wastes of thinning and maintenance cut left in the forest due to transportation problem and suitable for particle board production may be transported to factories after they are chipped with chipping machines in the forest, plantation of fast-growing tree species may be concentrated on, and production may be supported by renewing the technologies which get old on time. Pool and sprinkling system may be recommended for wooden raw materials in the facilities requiring long-term storing, by holding meetings between producers and users, reasons of the faults may be determined, it may be ensured that the mentioned faults are removed and common standard in the production is reached, and producers may be directed to agricultural forestry in order to produce raw material needed by their sectors. Industrial plantation works of both General Directorate of Forestry and private sector must be supported (OIAB sector report 2015).

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FRAUD CASES IN THE FIELD OF WOOD SCIENCE

Ioannis BARBOUTIS
Aristotle University of Thessaloniki, Faculty of Forestry and Natural Environment
University Campus, 54124 Thessaloniki, Greece
Tel: 0030 2310998895, E-mail: jbarb@for.auth.gr

Abstract

In recent years, there seems to be a significant increase of the cases of scientific fraud that are revealed in many scientific fields and in almost all the developed countries. In the specific work, an attempt is being made to present cases of serious evidence of research fraud in the field of wood science, in order to assist editors and reviewers in formulating appropriate quality principles to more effectively distinguish true research from false research. It is very difficult to determine if a research work is based on real experimental data, especially if there are not available basic information about the experiment carried out (place, laboratory, evidence of specimens or materials used etc.) and the respective authors (academic studies, scientific background, scientific cooperations etc.), and if there is no the contribution of all the scientists to this direction. The lack of specific information could lead the editors to accept an impressive fake investigation as real one and to encourage in that way, an increase in the number of such papers published. Offering the opportunity to be included in a publication as a co-author of someone that had not active participation in the research work should be always a matter of great concern, because it may conceal serious irregularities, fraud or even suspicious expectation of rewards in the future.

Key words: *fraud; wood science; editors; reviewers.*

INTRODUCTION

Generally, the fraud is widespread in all the human activities and it is mainly used for the acquisition of wealth and power. The first reference on fraud can be found in the Bible, where the "the Serpent" used lies to deceive Eve, while the first publication about the fraud was made by Lucian of Samosata in the 2nd century AD, which refers to Alexander of Abonoteichus, known as the false prophet. Lucian, in the introduction of his publication, stated that he feels shame to write such a work that it is dealing with the actions of a man, thrice cursed, who is not worthy to be read by educated people. This man, as Lucian says, excelled far beyond other people in cunning and intelligence. Furthermore, he had many other qualifications, but he used them for evil purposes. However, anybody who saw him for the first time had the impression that he was the most honest person. In fact, he apologizes to Pythagoras, for whom Lucian recognizes that he was wise and he had marvelous ideas, for his thought that if Pythagoras was contemporary of this Alexander, undoubtedly he would look inferior against him. Perhaps he is not totally unfair, since, as Xenophon mentions (4th century BC), most people usually agree with the bad guys and not with the remarkable people. According to Menander (4th century BC), this is because wicked men have the ability to use convincing and polite speech. It should be mentioned that Fraud comes from the Latin word fraus-fraudis, which derives from the Greek word φραδής (fradis), which means wise and shrewd (Liddell and Scott 1889).

Fraud in the science

Although scientists are considered to be the highest level of society and therefore, they should be an exemplar of honesty, they cannot escape their human nature and use often fraud tricks to increase and upgrade their published work. Perhaps, it would not be an overstatement to say, that scientist's familiarity with frauds begins from the period of their university studies with the "alleged" scientific education. A research based on 1800 students from 9 universities showed that 3/4 of them admitted cheating on tests or assignments (Fang and Casadevall 2013). The above research obviously emphasizes on the size of cheating by students. As it is in a race, where there is a winner, with the best qualities, and a loser with weaknesses, in the scientific fraud occasions, the responsibilities or reduced capacities of these professors should not be ignored.

A year ago the professor Lucio Picci (2016) of the University of Bologna announced that students are free to copy, since professors use to do the same. In this way, he wanted to publish incidents of plagiarism, where some of his colleagues were involved and who not only remained unpunished, because of the system that prevails within the university according to his statements, but also they were rewarded by being placed in senior positions. Unfortunately, this fact is not an

individual incident and of course, plagiarism is not the only form of fraud occurring in the scientific community.

Cases of scientific fraud have always been known, but have begun to seriously concern the general public towards the end of the 20th century, when published works on the extent of scientific fraud appeared, launching debates on what should be described as fraud (Hartemink 2000).

Generally, fraud cases encountered in scientific research are related to the fabrication of virtual-false work, falsification of existing publication results, dual publication and plagiarism by appropriating intellectual property of other authors (Rubin 2011).

A paper that have been reported as an example of fabrication (Hartemink 2000) is the "Petrol from plants", that was found that the results could not be reproduced. This paper was published on 1996 in the scientific journal "Nature" and it is still available to the audience at the price of 16€.

In 1998, after revealing that the data of at least one scientific paper of the Institute of Plant Breeding in Cologne was "fabrication", a team of scientists undertook an attempt to repeat the experiments described in more than 30 papers that started to be published from 1992 in some leading journals, such as Nature, Science, EMBO and PNAS. Already from the first test results, it appeared that many experiments were non-reproducible (Abbott 1998). A simple questioning on how ongoing fraud cases go unnoticed for so many years may not be enough, and apparently, such complaints may not have luck if it is to be examined by persons who may be involved in the specific or other fraud cases.

Another serious fraud case of several scientific papers falsification drew the attention of the German medical science from 1997 to 2005. A professor along with his female collaborator were accused for systematic falsification of data in many of their publications. The examination of 347 of the professor's publications showed that the 94 of them contained false data, and only 132 were free from any fraud. For this scientific work the Professor had received grants of hundreds of thousands euros, while the justice decided to stop the inquiry after an agreement of a 8000 euro payment by the professor (Tuffs 2004).

Technological development facilitated to a great extent the access and processing of electronic data resources, consequently another form of fraud, the one of plagiarism, becomes much easier. The fact of the information databases and sources increase that are available online make the detection of this fraud even more difficult. Although, techniques and software to facilitate plagiarism detection is in constant evolution-improvement, the intervention of a competent person is still required. Also, it would not be an exaggeration to be said that the discovery of a plagiarism case is usually a matter of chance (Clough 2000).

Although it is accepted that the negative impact of plagiarism and dual publication in science is not as important as the fabrication, such practices should be totally discouraged (Fang et al. 2012).

The examination of the withdrawal reasons for 2047 research articles on biomedical and life-science as indexed by PubMed revealed that 43.4% of withdrawals referred to fraud or suspected fraud, 14.2% were due to duplicate publication and 9.8% due to plagiarism. Referring to the geographical origin of the above research articles, most of them were in USA, Germany, Japan, China, UK, India and S. Korea with a slight variation in the order of the country depending in the type of fraud (Fang et al. 2012).

Several researchers have examined various cases of scientific frauds and have published works in order to highlight the existed problem and to contribute to the prevalence of genuine science. These publications cover various scientific fields but the field of medical science, according to number of publications, seems to have been more of a concern to the scientific community. Among these publications indicatively can be mentioned the books entitled: «Research fraud in the behavioral and biomedical sciences» (Miller and Hersen 1992) and «Fraud and misconduct in biomedical research» (Wells and Farthing 2008), the publications «Fraud in science» (Altman and Melcher 1983) and «Fraud and deceit in medical research» (Sarwar and Nicolaou 2012).

Similar publications, in a smaller extent, refer to fraud in different scientific fields such as organic chemistry (Rubin 2011) and archeology (Griffin et al. 1988). In all the fields of science there is the possibility some research fraud to have been committed. In some research areas, where experimental data are used, this possibility of fraud is more likely to be offered because of the obvious difficulty of immediate verification of the presented results. Theoretically, in some areas, such as computer programming, where there is a direct control of the proposed implementation the possibility of fraud may be significantly limited.

The main aim of publishing several works on fraud, apart from highlighting its extent and significance for the scientific credibility, is also to present proposals for a more effective control of scientific works before publication and the reduction of this phenomenon, which, as mentioned above, in recent years it is observed to continually increase. This impression may be partly subjective and

attributed probably to the ability of the easy access to Internet, as well the transmission speed of information, but it is evident that the abundant funding and widespread corruption usually stimulate scientific fraud to ease achievement of various personal ambitions. Based on the data published, the general opinion is that scientific fraud is carried out mainly by repeat infringers (Triggle and Triggle 2007).

OBJECTIVE

This paper aim to present, based on about 40 years of academic experience, cases that show serious research frauds in the field of wood science, in order to assist the work of reviewers and scientific journals editors in developing appropriate quality principles and standards for a more effective discrimination of actual research from the false or fraud research.

RESEARCH ON WOOD SCIENCE

Difficulties of Scientific Experiment

The research in wood science presents several difficulties. It is quite time-consuming, requires a lot of effort, it involves risks and there must be the appropriate equipment to carry out each test.

As an example of difficulty, it could be mentioned the relatively simple procedure of determination of the ultimate strength in static bending for a wood species according to ISO 3133: 1975. The process generally should include the following steps: selection and harvesting of wood material selected, transfer to the laboratory, sawing with band saw the plates, drying (time depends on the equipment and ranges between a few weeks to several months), resawing - planing shaping the final specimens to cross-sections of 2cmx2cm, with length along the grain of 300 to 380mm., thickness-width measurement of the specimens at the point where the pressure will be applied, determination of resistance to at least 10 defects free specimens in a testing machine, calculation of the static bending strength for each sample, calculation of the mean value of static bending strength and other statistical factors, cutting test pieces 25±5mm long from the point of rupture to determine the moisture content according to ISO 3130: 1975, weighing the test pieces and placing them in an oven at 103± 2^{o}C, cooling the test pieces in a desiccator, calculation of the moisture content for each sample and finally the mean value of moisture content. Even with the most up-to-date equipment, it is difficult for the whole process to end in less than 2 months, and the result that can finally be presented is just one number. It is obvious that a single number cannot express the overall effort that has been paid, nor can sustain itself a research work published. Many similar procedures are required to enable a work that can be accepted at a conference or a journal that does not have very high requirements. Also, the weight of the material, the cutting machines and the chemical reagents involve an increased risk of causing serious accidents, especially during the preparation stages of the experimental material. As an example of such an accident, it can be referred the crush of a foot toe of the writer of this work, caused by the drop of a relatively small piece of plane tree wood of the following dimensions: 5x10x50cm during the mechanical processing of the experimental material. Additionally, damages or unpredictable breakdowns of the laboratory equipment may seriously delay the completion of an experiment. Consequently, there are numerous difficulties in completing a scientific experiment and the results of the research may not be each time impressive, especially to the eyes of the journal editors, who look usually for very innovative, impressive and breaking-through results.

Documentation of research experiment

An essential prerequisite, however, for carrying out a research experiment on wood science is, as mentioned, the existence or accessibility and use of appropriate specialized equipment and, of course, the know-how of its operation. The absence of these basic operational research tools is obviously deprived of the possibility of anyone doing the research. The detailed description of the scientific area where a research experiment was carried out and the equipment used for this purpose is a particularly important element that should be included in each work because it provides the possibility of directly or indirectly verifying their existence. Including photographs from the conduct an experiment should be pursued as part of documentation for implementation. The publication of a research paper based on non-existent equipment should concern us not only for its validity, but also the reliability of the authors. The regular publishing of "Short Papers" types of manuscripts, which do not present in much extent the method and materials chapter should draw our attention. After a remark made to a very well-known journal of our field, commenting the tactics followed by "author X", the journal reported the following: "*It is of course hard to evaluate whether the papers of an author are based on real experiments or just smartly fabricated. Moreover, the peer-review process can only*

evaluate the plausibility of results, but not their actual origin. They will certainly consider it seriously if or when the same author submits a new paper".

Check of the publication reliability

The results of research in the wood sector contribute to the development of wood utilization but are generally not directly applicable and are not intended to meet particularly urgent human needs such as medical science. In general, the verification of their correctness is not regarded as an immediate imperative, nor their reliability can easily be denied especially when the results shown may range within reasonable limits, corresponding known results and thus appear to be plausible. Furthermore, it is obvious that it is practically impossible or at least very difficult to carry out the verification process, because it is essentially necessary to repeat the whole of a survey which, as mentioned above, generally presents several difficulties, and of course we could not have at our disposal material identical to what was investigated. However, wood as a biological material has a variable structure and quality, the characteristics and properties are influenced by many factors and therefore, a comparison even between the results of two experiments where similar conditions were used is very difficult. Generally, in all scientific fields, the check of the reliability of a publication is a difficult and time-consuming process, and the finding of whether it is the product of fraud, as mentioned earlier, or not is usually done after several decades (Griffin et al. 1988) or a random event (Clough 2000). In some cases, in wood science, photographic documentation of the experiment process and material could make a significant contribution to assessing the reliability of the research work. As an example of that, it could be referred a paper submitted to be published in a well-known journal dealing with particleboards manufactured from tree leaves and the only photograph included in the submitted manuscript was a simple tree leaf. As a reviewer, I asked a photo from particleboards constructed to be added. The answer was "*there are no available images from the produced particleboards and their tests*". The manuscript was rejected as suspected fraud, but it was published elsewhere, in 2 other journals, after minor changes in the title and different composition of authors.

The inconsistency of the results is a serious reason that should also draw the attention of the reviewers. For example, "author X" reports at least in 3 different papers in peer reviewed journals with a high impact factor, that he has determined sorption curves of wood specimens using different saturated salts for controlling relative humidity.The author reports the relative humidity for each of the six saturated salts he used (12, 23, 44, 55, 76 and 93%) but for the construction of the sorption curves he uses the RH values of: 10, 20, 30, 40 , 50, 60, 70, 80, 90 and 100. These values are not only different from the climates that correspond to the used saturated salts, but they are also more different climates than climates that the author reports that has used. The author goes further and produces models using these "findings" which seem almost perfectly fitted to the graph points.

Referring to a Higher Educational Technological Institute of Greece, related to Wood Science, the usual tactic that is followed, is to avoid choosing evaluators of their professors' work from the field of wood science, based on their specialization and knowledge, and instead, they prefer to choose evaluators from other scientific fields depending on how they will achieve better evaluation.

Papers manufacturing

Taking into account the long time that is required to conduct a research in wood science, it is clear that the number of research works a researcher can publish in a year is quite limited. Moreover, the required time for the writing and revision of the work according to the corresponding instructions provided in each case should be taken into account. A number of publications 2-6 per year, depending on the active participation of co-authors, perhaps could be considered reasonable by those who have conducted research in wood science. If it was stated that "author X" that he has managed within a period of 3 years (2006-2008) to publish 45 papers (which corresponds to 1 paper per 24 days), in most of them as the first author, we may have suspect or suppose that it is about journals or conferences proceedings without reviews. Adding the information that all the publications or presentations were peer-reviewed, and indeed many of them were in highly reputable journals such as Holz als Roh-und Werkstoff (8 publications) and Bioresources (3 publications), might well have led to the view that he is a scientist of extraordinary genius or perhaps something else may be happening.

The motivation for writing and publishing a research work, like almost all the human activities, mainly comes from the need to meet some indirect or direct financial goals. Young scientists seek to publish their work in order to enrich their curriculum vitae so that they can earn or improve a job with the best possible financial gain. Also, a good resume can significantly increase the chances of approving and funding a research proposal.

Who is who

Each author's main goal is to publish his research work in a well-known international journal with the highest possible impact factor in order his work to be considered of better quality. However, the success of publishing in a good journal, as has been shown in scientific fraud cases, should not guarantees us the validity of the content of the publication paper. By publishing a paper, everyone can access its content, but usually there is very little or no information on the contributors of work, which is a very important criterion for assessing the quality and authenticity of the research being carried out. For example a young writer is expected to be personally known, only to few scientists in the industry or academic world, but with the development of electronic media, it is likely through his published works, his name to become known very fast to many scientists all around the world, but only as a name, not personally. By increasing the number of his publications, his name will begin to gain prestige in the scientific field and improve his circle of acquaintances. However, substantial information about the level and type of studies, education, workplace, collaborators, and other curriculum data that can outline the personality of the authors and assess their contribution to the completion of a scientific work are generally still very limited. It should not be considered an exaggeration the thought that, despite the widespread dissemination of information technology and the possibility of getting information even for the most insignificant things, there are instances where we cannot have fundamental information about what we believe to be the elite society. Of course, the lack of specific information for an author make us not to have reasons to consider him unreliable. As an example, it could be mentioned the absence, until a while ago of an English-language Curriculum Vitae of "author X", one of the most well-known international Greek writers in the field of wood science, with more than 700 international bibliographic references, with a total impact factor of 62.65 and h index 15, his CV was only available in the Greek language. Reading his CV reveals that his total time spent in higher education (not university) up to his doctoral degree, was around 6 years. Perhaps, it is a unique phenomenon not only in the field of wood science, but for the whole scientific world. A more detailed examination of his resume shows that most of the period of the 2 years he needed to obtain his doctorate, he was working at the same time on a different subject in another country. In addition, there is the possibility that some of the information that someone mentions in his CV not to correspond to reality. In Greece, it is widely known the phrase "you are, what you state that you are" and this should not be considered as an exaggeration, as there were often known cases of people who have been selected in important public positions with false education documents. But in a period of globalization and commercialization of education, the responsibilities extend beyond the national boundaries of a country. In a question at a University of Great Britain about the legality of granting a doctorate to "author X", based on the data he presents in his resume, the answer was that «*We are unable to confirm any further details with you regarding this matter*». A similar question posed to the highest competent Greek audit services regarding the legality of recognition of the above doctorate was never answered, probably because of the participation of a high-ranking political figure in this process. It should be noted that "author X" already works as a professor at a higher education institution of Greece and in addition, several of his co-authors, have taken advantage of this works to improve their academic status.

According to the scientific ethics, as well as simple logic, it is legitimate and expected that each scientist should deal with the subject matter he has specialized in, since scientists should not talk on matters that are not enough aware of and specialized on, such as the example of a doctor that can not give lectures and analyze topics of Astronomy. As mentioned previously, a publication only mentions the name of the author and does not refer at all to his scientific background, which should constitute the most important indicator of scientific competence and qualitative assessment of a research work. Presenting this information may overturn the expectations and reliability of a publication. In a paper recently presented at a conference in Zagreb entitled "Particleboards with Wood in Various Forms", participated as a co-author a professor with specialization in: "Semiconductor Electronic Properties, Semiconductor Devices and Thermal Analysis of Materials". Of course, his specialization could possibly be of interest to the participants in various ways.

A professor in the field of Wood Science at Higher Educational Technological Institute of Greece, justified the suitability of a candidate, with a Doctoral Degree in genetic diseases, to a position of wood science saying the following: "*His doctoral dissertation deals with organic substances, as well as wood, which is also an organic substance, therefore it has relevance to the subject "Chemical wood treatments*».

Furthermore, even in our Faculty, there is a case of a scientist with an official scientific specification of "Mountainous Water Management", who was considered to be suitable to work on the field of Wood Technology.

Co-Authors

Although the completion of a scientific publication, in the majority of fields of wood science, for an intelligent and knowledgeable scientist of the field, is possible to be implemented by individual effort, partners' involvement can make a significant contribution to reducing the time required and improving the quality of work or provide higher prestige. Since, the presentation of the field or the degree of participation of each partner is not required to complete the publication, it is therefore logical that all participants have the opportunity to enjoy almost equally the benefits of a publication even if not they were not actively involved in it. "Author X" has also presented in at least 12 papers his wife as a co-author, a graduate of a technical institute, without any specialization in the science of wood. But a real researcher, like any worker, apparently would not wish to share the performance of painful efforts to someone who has not had the slightest involvement in a task. Honorary participation in a publication as authors of people who did not have an active participation in it, should raise reasonable questions about its credibility, correlating it to the phrase of Virgil "timeo Danaos et dona ferentes" (Beware of Danae and bearing gifts). This is because the basic problem that a pseudo-researcher is concerned with is not whether he will share his non-existent effort with others, but how he can best document his work as true. Adding one or more names as co-authors makes the research work appear as a result of a research team collaboration, while helping the paper to be highly evaluated and be exploited in various ways, especially when it is possible to involve well-known scientists or high-ranking individuals. It could be compared with a brilliant package that has the ability to upgrade the aesthetic value of a gift of very low value. In a question I posed to a co-author of "author X" about the level of his participation in the work referring to "bonding behavior of wood particles", which was published in Holz als Roh- und Werkstoff journal, I received the much honest answer, that his name was included only "as an honour (*honoris causa*)" (not at all working). Co-authors from different countries may theoretically increase the importance and prestige of a work, but should raise questions about how active such a research cooperation could be. For example, in a work on the experimental measurement of Soil Compaction and Porosity Changes in a forest of Northern Iran, it is certain that at least one of the co-authors never visited Iran. The excellent knowledge of the English language or the literary skills that one has and contributes to the writing of a work should not be a reason for accepting him as a researcher.

CONCLUSIONS- PROPOSALS

Cases of scientific fraud are constantly revealed, especially in scientific fields where the interest is attracted and of course where there can be achieved ample funding. Most cases seem to come from the most developed countries and from scientists who were considered highly successful in their field. It would be unrealistic to argue that there are no cases of fraud in the field of wood science. But the extent of it may be difficult to be assessed. Most, if not all wood scientists, may have experienced cases of scientific fraud. Some of them have already accepted to engage in a work as co-authors "*honoris causa*". It is very difficult to determine if a research work is based on real experimental data or not, especially if basic information for the author, the cooperation and the experiment are not available or if there is not the subscription of all scientists to that. By staying inactive or maintaining opaque procedures we contribute to increasing this phenomenon or perpetuating false impressions.

For a more qualitative and valid assessment of a research work and the ensuring of the good fame of the scientific world, it would be useful to introduce, in addition to the rules already applied, some additional simple requirements that could contribute to the safer traceability of a work's, as well as the author's credibility. For this purpose, it is useful to highlight the following:

- The authors should refer to their specialty, their workplace and the site from which more information can be obtained.
- To justify the role of participation of each co-author.
- A more detailed description is required in the experimental methods and materials used and emphasis should be placed also on the equipment used and the place where this equipement was available.
- It would be particularly important, the presentation of the experiments to be accompanied by relevant evidence, such as photographs of them.
- Educational and research institutions should contribute to information sharing and meritocracy, avoiding mysticism.
- Journals should cooperate in order to establish a common open information base-system for authors, universities and research centers accused of scientific fraud.
- The reviewers should look for and focus more on evidence that proves the credibility of an research work, but the editors should also pay more attention to the selection of the reviewers.

- Remember that, offering the chance of participation of our name as co-author in a scientific publication without any active participation, may hide fraud case and may conceal serious irregularities or reward expectations.

It is obvious that we do not live in an angelically made world, and therefore fraud undoubtedly will continue to exist. However, this does not mean that the constant improvement of practices for the discovery and limitation of scientific fraud should stop. It is disheartening, especially for young researchers, when the scientific work based on laborious laboratory research is being overshadowed by publications based on fraud methods.

It is necessary to take into consideration the fact that a scientist who possess fake diplomas or has achieved possessing them through the use of fraudulent means, is expected that he will not have nor reliable scientific research work, neither could consist a meritocratic reviewer.

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ECONOMICAL ENGINEERING ASPECTS APPLIED IN THE MANAGERIAL DECISION FOR THE IMPLEMENTATION OF AN OPTIMIZATION METHOD TO STREAMLINE THE PROCESSES IN THE WOOD INDUSTRY

Radu Dan PALTAN
Transilvania University of Brașov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0745823722, E-mail: radu.paltan@unitbv.ro

Loredana Anne-Marie RADULESCU
Transilvania University of Brașov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0722953653, E-mail: loredana@unitbv.ro

Cristina Maria BIRIS
Lucian Blaga University of Sibiu, , Faculty of Engineering
Bulevardul Victoriei 10, Sibiu 550024, Romania,
Tel: 0269 216 062, E-mail: cristina.biris@ulbsibiu.ro

Abstract

The quality and cost of products in general and of wood products in particular is a crucial condition both in selling them on the market and also in attracting buyers and satisfying their desires and exigencies.

The quality and value of the products depends heavily on the technical performance of the equipment, the skill level of the workforce, the organization of the manufacturing and processing processes.

A higher quality level can be achieved by eliminating as much as possible elements that negatively affect yields, raw materials, materials, and energy consumption. Some of them lead to the need to re-technologize the production lines with the ultimate effect of raising the quality of the products from a technical point of view.

Evaluations can refer to both the technical parameters and the economic or combined parameters to facilitate process management, positively influencing both the loss of material, energy, but also the growth of the company's profit. Following the research carried out, the authors of this paper have found technical and economic solutions that contribute to the optimization of the company's activity.

Process optimization can be done by applying different methods to evaluate the performance of the studied process, such as: Target-Costing Method; Taguchi method; Six Sigma Method; and so on.

Key words**:** *optimizing; technological streamline; efficiency; management; production.*

INTRODUCTION

The grinding process, which occupies a significant percentage of the products in this company, has been studied. It is linked to the milling process that has to be optimized and one of the important efficiency criteria is energy consumption. The most eloquent parameter for wood milling that has a major influence on energy consumption is Wood Specific Resistance or Specific Cutting (K), which characterizes the processed wood material and which can be calculated based on the absorbed power (equation 1):

$$P = f(K) \Rightarrow K = f(P) \tag{1}$$

Knowing that:

$$P = \frac{K \cdot b \cdot h \cdot u}{6 \cdot 10^3} \tag{2}$$

In which: P = cutting power, measured in kW;
K = specific wood resistance daN / mm2 or specific workpiece [daNm / cm3]
B = cutting width, measured in mm;
H = cutting depth, measured in mm;
U = feed rate, measured in m / min
It results that the specific resistance is directly proportional to the absorbed power and can be calculated with the formula 3:

$$K = \frac{P \cdot 6 \cdot 10^3}{b \cdot h \cdot u} \text{ [N/mm}^2\text{]} \quad (3)$$

OBJECTIVES

Optimization criteria once applied to the studied process will inevitably lead to finding technical solutions to meet the requirements of the market. Optimizing a process can be done by applying various methods of assessing the performance of the studied process, such as the Target Costing method, the Taguchi Method, the Six Sigma method and so on.

As a result of the unfolded researches, the authors of this paper have found the answer to two questions(which of the applied assessment methods are most effective? and which of them would lead to those improvements that converge towards a real optimization?) an answer that will be detailed in the presentation below.

METHOD, MATERIALS AND EQUIPMENT

It is known that in the cross-sectional processing of the wood material compared to its longitudinal directional processing it is noticed a significant difference in the energy consumption. The veracity of this statement is also confirmed by the values obtained for the cutting power required for the transverse and longitudinal cutting of the wood in the experimental researches [..] and which have been recorded in the table (Table 1). The processed data were represented graphically (Figure 2a and 2b)

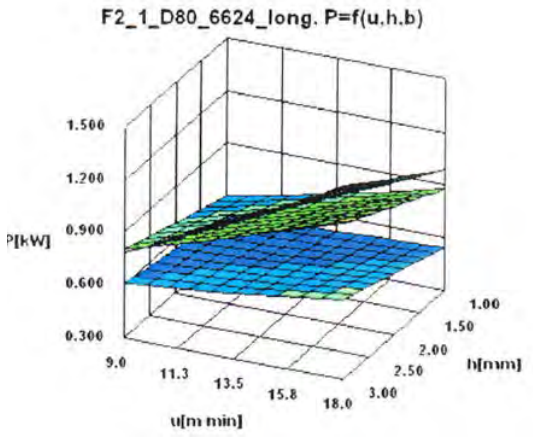


Fig. 1.

In cross-sectional wood processing, apart from the high energy consumption difference with respect to longitudinal section milling, the processing technology also requires additional machining phases in order to finish the products of the same quality as the processing of the wood after processing in the direction longitudinal.

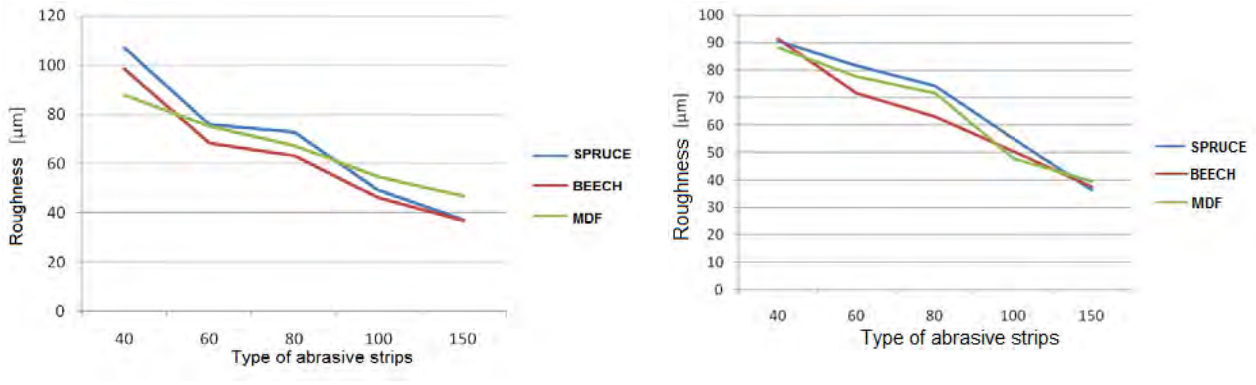
These steps are generating additional costs and are also responsible for prolonging production times. At the same time, more waste is generated during the additional finishing phases and can increase the risk of generating more scrap (needing more complicated models of manual finishing).

The quality of the product or process is indirectly proportional to energy consumption and power absorption. So:

Table 1

Values of power consumption and amount of dust resulting from grinding

Grit	Depth of cutting [mm]	Type of wood	Advance speed (*u*) 4,5 m/min (*Pa*) Average power consumed at cutting [kw]	Advance speed (*u*) 9 m/min (*Pa*) Average power consumed at cutting [kw]
P40	0,4	Spruce	4.687935	7.168482
	0,4	Beech	8.298308	11.20128
	0,4	MDF	2.150012	2.61206
P40	0,3	Spruce	4.687935	6.820808
	0,3	Beech	6.097583	10.15306
	0,3	MDF	2.471298	4.103166
P60	0,4	Spruce	5.73899	7.547807
	0,4	Beech	8.884849	N/A
	0,4	MDF	4.523018	5.117319
P60	0,3	Spruce	4.085989	6.739016
	0,3	Beech	6.444596	11.60971
	0,3	MDF	2.738578	3.119402
P80	0,4	Spruce	6.347257	7.995917
	0,4	Beech	9.693495	N/A
	0,4	MDF	3.134177	3.170587
P80	0,3	Spruce	5.06474	7.634242
	0,3	Beech	7.712464	11.86412
	0,3	MDF	2.023131	3.323742
P100	0,3	Spruce	4.858144	7.749881
	0,3	Beech	7.94103	11.98587
	0,3	MDF	1.777011	4.610409
P150	0,3	Spruce	6.744672	10.67864
	0,3	Beech	9.906182	N/A
	0,3	MDF	4.993595	6.216813



a b

Fig. 2.

a- Graphic representation of roughness values of the Rz parameter, in the grinding transverse direction with a feed rate of 4.5 m / min, for 5 types of abrasive strips (P040, P060, P080, P100, and P150); b- The roughness values of the Rz parameter in a direction perpendicular to grinding, depending on the feed rate of 9 m / min and the abrasive tape (Darii 2011).

From the multitude of techniques and methods of economic analysis used to optimize production costs, following a meeting with the company management together with the engineers directly involved in production, we reached the conclusion of using the Multicriteria Analysis method in which the following three Methods for Economic Efficiency of SC Famos SA:

The Target Costing method: is part of a strategic management approach according to which each cost carrier is analyzed throughout their life cycle. The target or objective cost is a cost management concept used and developed in Japanese companies, especially in the automotive industry since the 70s. Underlying this target cost concept there was the need to produce smaller series of products that can better adapt to the market needs, the introduction of the new production organization methods (Just In Time operating system) and the introduction of automation-based technologies (CIM - Computer Integrated Manufacturing - systems).

The Taguchi method imposed itself as being more effective than other methods of experiment planning. It derives from the method of factorial experiments and proposes an alternative method for calculating the average effects of factors and interactions, thus making mathematical modeling much easier. Its efficiency is due to reducing the number of tests given by the split plan method, which allows modeling with much less experiments than the full plan method. The Taguchi method is one of these split plan methods and has also the advantage that it is easy to apply in practice. When determining the number of experiments involving the study of a phenomenon by this method, stricter conditions must be applied.

The Six Sigma Method represents the standard deviation in statistics and it is a management methodology aimed at increasing product quality by determining and removing the continuous causes of defects and process variability (potential or detected) in order to ensure customer satisfaction, based on the methods FMEA (Failure Mode and effect and Analysis) and QFD (Quality Function Deployment) and modern management methods applied to the joint teams made up of manufacturer, supplier, client, professional, research centers etc.

Each of the three methods aims to achieve the economic efficiency of the company by taking into account certain parameters that significantly or less significantly influence the process optimization. Among the projected (common and / or different) parameters for each method, there were studied only the parameters that the three methods have in common, so that a managerial decision can be taken on the method to be applied within the company.

The optimal decisions for the company management, since there are several alternatives (methods), can be obtained by applying a multi-criteria analysis method (AMC). The goal of this method is to conduct a comparative evaluation of the proposed options (methods). For the AMC, all common parameters of the selected methods were taken into account simultaneously in a complex situation. The method is designed to assist decision makers by integrating different options, reflecting the views of the actors involved in a prospective or retrospective framework. The analyzed parameters reflect how objectives are achieved. The best option will be the one that will be closest to achieving most objectives and that will obtain the highest scoring.

The comparison was made between the efficiency models that could be selected taking into account the specific characteristics of the wood processing industry.

The analysis consists of the calculation of value hierarchizing coefficients, for the performance of the objects under comparison. The result of the multi-criteria analysis, properly applied, provides scientific and effective results for optimal solutions in the technological process which will be reflected in the economic cost/price of the final product.

RESULTS AND DISCUSSION

The multi-criteria analysis (MCA) describes any structured approach to be used in determining the general preferences of several alternative options, options that lead to achieving a number of objectives. In the current case, our main goal is to optimize production by reducing costs at SC Famos SA. The steps that were followed are:

1. Establishing methods and evaluation criteria in the decision-making context.

For identifying the optimization method with the greatest applicability in the wood processing industry, the research was based on the use of brainstorming techniques and multi-criteria analysis.

Within this step there were identified the alternative criteria to be taken into account. In our case the options considered are: alternative 1 the Target Costing method; alternative 2 the Taguchi method, alternative 3 the 6 Sigma method.

Through the multi-criteria analysis (MCA) there will be eliminating the subjectivity, since the order of the criteria is determined by comparing each criterion relative to other selected criteria.

The brainstorming consisted in organizing a work meeting with a group consisting of the company management and 10 engineers from SC Famos SA Odorheiul Secuiesc. During the brainstorming session, held in the company's meeting hall, there were presented several ways to optimize production from which there were chosen the 3 mentioned methods and the participants generated a number of ideas regarding the applicability of these methods in the technological process. After continuing the brainstorming session, there were generated following evaluation criteria that must be followed in the process of multi-criteria analysis:

1. Cost of implementing the methods (C);
2. The simplicity of using the method (S);
3. The effect of applying the method reflected in a reduction of the energy consumption (E);
4. Acceleration of the technological process - eliminating some steps in the process => reducing the time and costs that are reflected in the price (A);
5. Ease of implementing the methods (U);
6. The quality of the product (A);
7. Number of failures / waste (N).

2. Determining the share of criteria common for the three methods selected for relative quantifying. At this stage, after identifying the 7 common criteria which are relevant for solving the problem in selecting the decisional problem for the optimization method, they have been ranked. This ranking took into account the major categories of costs and benefits resulting from the options considered. The scoring was done according to the principle – a criterion is more important than the other (= 1), as important (= 1/2), less important (= 0)

Table 2 presents the calculation of points, the level and the weighting coefficient Yi.

Table 2

Point calculation, the level and the weighting coefficient Yi

Criteria	C(1)	S(2)	E(3)	A(4)	U(5)	O(6)	N(7)	points	level	Yi
C(1)	0.5	0	0.5	0	0	0.5	0	1.5	7	0.27
S(2)	1	0.5	0.5	0.5	1	1	0	4.5	2	2.89
E(3)	0.5	0.5	0.5	0	0.5	1	0	3	5	1.17
A(4)	1	0.5	1	0.5	1	0	0	4	3	2.2
U(5)	1	0	0.5	0	0.5	0	0.5	2.5	6	0.77
O(6)	0.5	0	0	1	1	0.5	0.5	3.5	4	1.64
N(7)	1	1	1	1	0.5	0.5	0.5	5.5	1	4.57

3. Hierarchizing the options. As a result of the inter-criteria comparison process, there resulted a matrix whose sum by lines (sum of each criterion in part) (column 9) resulted in a hierarchy (column 10) of the criteria's significance as seen in table 2.
The next step in the MCA procedure was calculation of the weighting coefficient (Yi) column11, which is calculated using the FRISCO equation (4):

$$\gamma_i = \frac{p + \Delta p + m + 0{,}5}{-\Delta p' + \frac{N_{crt}}{2}} \tag{4}$$

where:
p - is the sum of points obtained on the line by the considered item;
Δp - the difference between the score of the considered item and the score of the item at the last level.
m - represents the number of surpassed criteria, i.e. the number of criteria with scores below the item;
NCRT - number of criteria taken into account;
Δp` - the difference between the score of the considered element and the score of the element in the first rank (a negative result).

4. The standardization of scores for each criteria was made in a common scale ranging from 1 to 10 and taking into account the effect that it will have on the company's activity. The result of the analysis is presented in Table 3.

Table 3

Criteria and their value range

CRITERIA	INDICATOR	VALUE RANGES	EFFECT
C1(C)	1. Cost of implementing the methods (C);	1-10	-
C2(S)	2. The simplicity of using the method (S);	1-10	+
C3(E)	3. Effect of applying the method reflected in a reduction of the energy consumption (E);	1-10	+
C4(A);	4. Acceleration of the technological process (A);	1-10	+
C5(U)	5. Ease of implementing the methods (U);	1-10	+
C6(O);	6. The quality of the product (A);	1-10	+
C7(N).	7. Number of failures / waste (N).	1-10	-

5. The creation of the performance matrix describes the expected performances of each option according to the chosen criteria. The information on the dimension of each criterion can be expressed in units. Table presents the performance matrix and the assigned values were determined by the personnel of SC Famos SA within the organized brainstorming session

Table 4

Performance matrix

	TC Method	T Method	6S Method
C(1)	8	9	10
S(2)	9	8	10
E(3)	9	10	9
A(4)	10	8	9
U(5)	10	9	10
O(6)	10	10	10
N(7)	9	10	9

6. Examining of results. Considering the performance matrix and the weighting coefficient there was calculated the product between the Yi coefficient and the performance degree Ni, then the products

were added by criterion to achieve the final result. The data obtained are presented in Table 5 and can be compared.

Table 5

Calculation of the products between the grades N and the weighting coefficients

		Method TC		Method T		Method 6S	
Criteria	**Yi**	Ni	Ni*Yi	Ni	Ni*Yi	Ni	Ni*Yi
C(1)	0.27	8	2.16	9	2.43	10	2.7
S(2)	2.89	9	26.01	8	23.12	10	28.9
E(3)	1.17	9	10.53	10	11.7	9	10.53
A(4)	2.2	10	22	8	17.6	9	19.8
U(5)	0.77	9	6.93	9	6.93	10	7.7
O(6)	1.64	10	16.4	10	16.4	10	16.4
N(7)	4.57	9	41.13	10	45.7	9	41.13
Final Score			**125.16**		**123.88**		**127.16**

The final analysis was done taking into account the sums of all criteria that influence each method. It can be noticed that 6S is the most effective method, totaling the highest value.

In MCA procedure, there is also a sensitivity analysis stage that was not required to be unfolded here, given that the sensitivity of the criteria in relation to changing the method is very close.

CONCLUSIONS

As a result of the multi-criteria analysis applied in the wood domain at SC Famos SA in order to choose the evaluation method for the optimization of the technological flow, the result is that the most efficient method that the company management has to choose for its application in production is the 6 sigma method Achieved the highest score of 59.61 against the Target Costing method which achieved 58.38 and the Taguchi method which only achieved 56.33

These results confirmed the discussions and opinions of the engineers at SC Famos SA during the brainstorming session that confirmed that the Taguchi method is an obsolete method due to the managerial principles that takes them into account and the Target Costing method is limited to its application to certain industrial branches Being incomplete in relation to the many aspects and management decisions that management has to take into account in the woodworking industry.

Following the application of the 6S method to optimize the cutting process found in SC Famos SA's manufacturing technology and which mainly influences production, one of the criteria was energy consumption. The most eloquent parameter for energy consumption in wood milling is the specific resistance (K), which characterizes the processed wood material and which, when milling, can be calculated according to the absorbed chipping power.

It has been found that a significant difference in the energy consumption is recorded in the processing in the transversal direction of the wood material compared to the longitudinal direction of its processing, the power being consumed being much higher in the first case.

In order to achieve minimal thickness variability and a minimization of waste in the case of an even wood lamella, the feed rate of the wood in the class varies according to the classes, which will be the subject of further research.

Also, in cross-sectional processing, besides higher energy consumption, it also requires additional processing phases, in order to finish the products obtained with the same quality as the woodworking in the longitudinal direction. These steps are generating additional costs and are also responsible for prolonging production times. At the same time, additional waste generates more waste and can increase the risk of generating more scrap (needing more complicated manual finishing).

Based on the results of the study, the company implemented a process of wood equalization before the cutting process and is ready to make refurbishments for a start on a production line, the first stage was the acquisition of a new saw blade machine to optimize the process equalization of wood.

The authors of this article are currently preparing to evaluate the new process, namely, to investigate process capacity, optimal debit rates, and assess the level of material losses after implementing the proposed changes.

Despite the slightly higher costs to implement the management decision through the 6 sigma method noted in the implementation, they will finally be annihilated and will have the effect of cost optimization by minimizing the volumes of waste and waste produced.

A unanimous opinion was on the ease with which this method can be applied at all stages of production, especially as it involves the involvement of workers in the measurement, evaluation and continuous improvement of parameters.

The methodology presented for the optimization of technological processes to make the furniture production process more efficient is universal and can be applied to other similar processes in the wood industry (wood panels, parquet etc.).

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SECTION 12. POSTERS

STRUCTURAL CHANGES ON WOOD SURFACE AND THERMAL PROPERTIES EVALUATION AFTER CHEMICAL TREATMENT

Carmen-Alice TEACĂ
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
E-mail: cateaca@icmpp.ro

Liliana ROŞU
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
E-mail: lrosu@icmpp.ro

Cristian-Dragoş VARGANICI
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
E-mail: varganici.cristian@icmpp.ro

Dan ROŞU
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
E-mail: drosu@icmpp.ro

Fănică MUSTAŢĂ
Physical Chemistry of Polymers Department
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
E-mail: fmustata@icmpp.ro

Teodora RUSU
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
E-mail: rusu_teodora@yahoo.com

Abstract

The paper presents the results of an experimental study performed using a softwood species - fir wood (Abies alba L.)-, originating from mature trees. Dried wood samples as discs were treated with organic anhydride in the presence of solvent using different concentration levels. The extent of wood treatment was determined by calculation of weight percent gain values, these increasing from ~ 11% up to 39%, depending on the organic anhydride concentration. Structural changes occurred on the wood surface were investigated by Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) analysis. FTIR spectral changes evidenced wood modification by reaction with organic anhydride in the spectral region (1739 -1724) cm^{-1}. XPS investigation quantified changes by the increased atomic ratio of oxygen to carbon (O/C) due to the oxidation process occurred during wood modification process and with a detailed analysis of the contributions to the C1s peak in spectra. Thermal stability was also investigated by thermal analysis (TGA). Wood samples presented better thermal stability after chemical modification with increasing organic anhydride concentration and when comparing with initial wood.

Key words: *softwood discs; fir; chemical modification; structural changes; thermal stability.*

INTRODUCTION

Wood represents a versatile multi-component polymeric system, with many potential applications, being renewable, biodegradable and susceptible to structural changes. The surface of wood deteriorates relatively fast when wood is exposed to the environment without any protection (Hon 2001; Hon and Chang 1984; Pandey 2005; George et al. 2005; Müller et al. 2003). The occurred dimensional changes are a consequence of moisture and drying. Roughness of the wood surface is enhanced through the leaching of photo-degraded wood fragments (derived mainly from lignin) by rain water, thus enhancing and consequently exposing underlying cell wall layers to further erosion processes.

Protection of exterior wood from deterioration can be ensured by applying two effective measures, namely surface coating and bulk chemical treatment of wood which can act individually or conjointly. Coatings protect the wood surface against harmful environmental factors and decay by fungi. Transparent coatings need additional of UV absorbers and radical scavengers for their protetction, as well as for the wood surface (Ahola 1991).

Chemical modification confers wood dimensional stability, either by deposition of chemicals in the wood mass, or by cross-linking wood fibers with polymers (Chang and Chang 2006; Hill et al. 2000). Acetylation is also efficient in improving coating performance (Evans et al. 2000). When cyclic anhydrides are used for wood chemical treatment, ***e.g.*** succinic anhydride, the carboxylic acid formed through esterification reaction is now attached to the wood, as presented in Fig. 1. Nevertheless, an extra cross-linking process can stabilize wood materials even better, besides improving the hydrophobic properties. Thus, wood may exhibit a good water resistance alongside an improved dimensional stability and decay resistance (Rowell 2006).

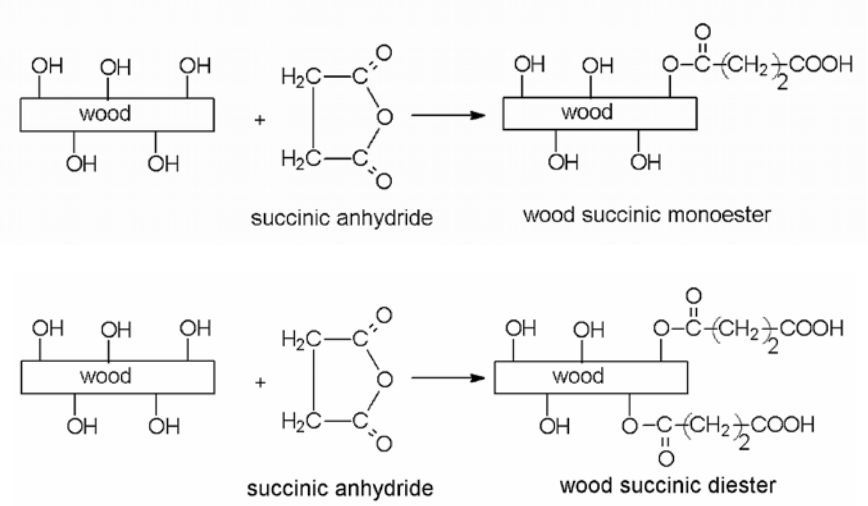

Fig. 1.
Chemical modification of wood samples - Possible reactions.

Structural changes occurring on wood surface after chemical modification may be investigated using Fourier transform infrared spectroscopy (Roşu et al. 2010; Colom et al. 2003; Pandey 1999), and X-ray photoelectron spectroscopy (XPS) methods. XPS is a non-destructive surface analytical technique providing information on the oxidation or chemical bonding state of elements and successfully used in several applications related to the wood science including characterization of chemistry for different wood species (Bouafif et al. 2008; Nguila Inari et al. 2006; Nzokou and Kamdem 2005; Sinn et al. 2001), chemically modified pulp and wood (Prakash and Mahadevan 2008; Matuana et al. 2001; Chtourou et al. 1995), surface lignin on cellulose fibers (Johansson et al. 1999), and weathering of wood and wood–plastic composites (Matuana and Kamdem 2002).

OBJECTIVE

The main objective of the present research was to evaluate the structural changes occurred on the wood surface after chemical treatment of a softwood species with organic anhydride using Fourier transform infrared spectroscopy (FTIR) and X-ray photo-electron spectroscopy (XPS) methods. This evaluation was performed by comparison with initial wood samples, without treatment. Thermal properties were also considered and investigated by simultaneous TG-DTG analysis.

MATERIAL, METHOD, EQUIPMENT

Wood specimens as discs were prepared from logs of softwood *Abies alba* L. previously dried at ambient temperature for one year. Wood samples were polished with sandpaper (400 P) prior use. After removal of extractives using solvent extraction in a Soxhlet apparatus, wood samples were subjected to the drying process for 24h in an oven at 70°C until a constant weight was reached. Reaction with succinic anhydride (SA) as solution in solvent with different concentration levels (60%, 80%, and 120% respectively expressed as w/w) was performed for 60 min under continuous stirring at 100°C. After reaction end, wood samples (10 samples for each concentration value, R reference samples) coded as MW(SA60), MW(SA80), and MW(SA120), respectively, were taken from the solutions, maintained at room temperature for cooling, prolonged washed with solvent for removal of non-reacted organic anhydride, and finally vacuum oven-dried for 24h at 70°C to reach a constant weight. The extent of reaction was calculated as weight percent gain (WPG) determined by the differences in oven dry weight of the sample before modification (W_1) and after modification (W_2) according to the equation [$WPG = (W_2–W_1)/W_1 \times 100$].

FTIR Spectroscopy

FTIR spectroscopy of wood samples was conducted on a Bruker FTIR spectrometer (Vertex 70) with a MIRacle ATR accessory for single or multi-reflection attenuated total reflectance. The single-reflection sampling plate of the accessory has a 1.8 mm round crystal surface allowing reliable analysis of small samples. The ATR crystal plate is from Diamond, and solid materials can be put into intimate physical contact with the sampling area through high-pressure clamping, yielding high-quality, and reproducible spectra. Spectra were registered in the range from 400 to 4000 cm^{-1} at a spectral resolution of 4 cm^{-1} and 64 scans were averaged. The bands from FTIR spectra for wood samples were attributed with the aid of the literature data (Colom et al. 2003; Owen, and Thomas 1989).

X-ray Photoelectron Spectroscopy Analysis (XPS)

Surface characterization of wood samples was conducted on a KRATOS Axis Nova spectrometer (Kratos Analytical, Manchester, UK), using Al-Ka radiation, with 9mA current and 15 kV (135 W), and base pressure of 10^{-8} to 10^{-9} Torr in the sample chamber. The incident mono-chromatic X-ray beam was focused on a 0.7 x 0.3 mm^2 area of the surface. The XPS survey spectra for the samples were collected in the range of -10÷1200 eV with a resolution of 1 eV and a pass energy of 160 eV. The high-resolution spectra for all the elements identified from the survey spectra were collected using pass energy of 20 eV and a step size of 0.1 eV. The binding energy of the C_{1s} peak was normalized to 285 eV. Data were analyzed using the Vision software from Kratos (Vision 2.2.10).

Thermal Analysis (TGA)

Simultaneous thermogravimetry (TG) and differential thermogravimetry (DTG) were performed using a thermal analyzer STA 449 F1 Netzsch (Germany). Samples (≈ 5 mg) were placed in Al_2O_3 crucibles, and heated under nitrogen flow from room temperature up to 600°C at the 10°C/min rate of temperature.

RESULTS AND DISCUSSION

Extent of Wood Chemical Modification

The time of reaction has a significant role on the values of WPG, and many of the properties of modified wood samples depend on the applied treatment. The amount of moisture present in the wood and wood constituent polymers is also important. The content of moisture (~ 5%) seems to be needed for best reaction, but above this level the water hydrolyses succinic anhydride to corresponding carboxylic acid. This loss by hydrolysis accounts for a 5.7% loss of anhydride with each 1% of water present in the wood structure. The rate of modification reaction decreases with increasing moisture content. Generally, an increase of SA concentration determined an increase in WPG in the range 11-39%. The maximum WPG was obtained when wood sample was treated with 120% (w/w) SA.

FTIR Spectroscopy

FTIR spectra recorded for both non-treated and treated wood samples are presented in Figs. 2-5.

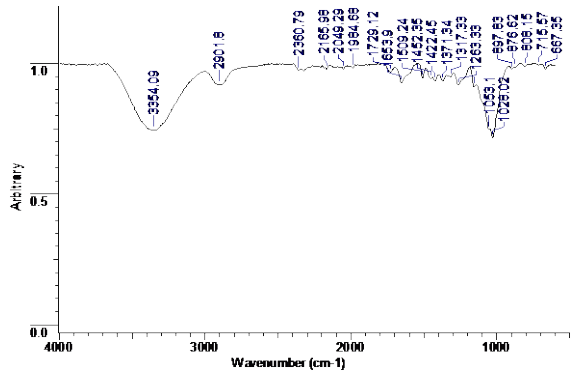

Fig. 2.
FTIR spectrum for reference (R sample).

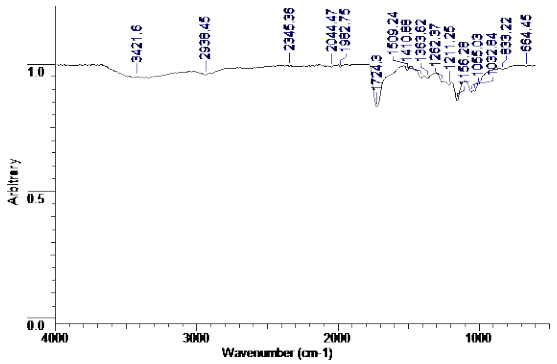

Fig. 3.
FTIR spectrum for MW(SA60) sample.

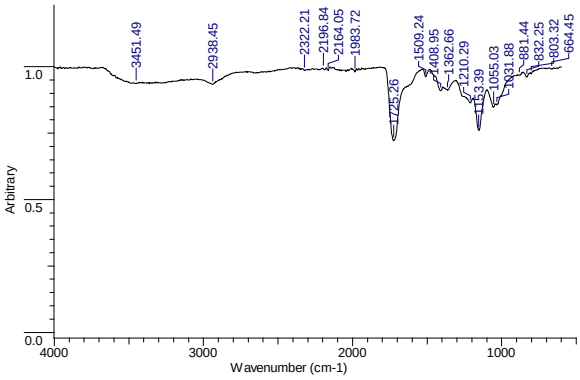

Fig. 4.
FTIR spectrum for MW(SA80) sample.

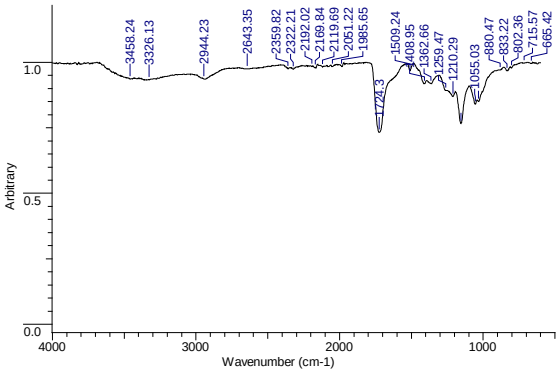

Fig. 5.
FTIR spectrum for MW(SA120) sample.

A decrease in the intensity of the O–H absorption band (mixed hydroxyl groups originating mainly from cellulose and hemicelluloses) at 3364 cm^{-1} is observed, indicating that the hydroxyl group contents in wood are reduced after chemical modification reaction. A stronger carbonyl band at 1725 cm^{-1} is noticed for chemically modified wood with increasing SA concentration. The enhanced carbonyl absorption peak at 1725 cm^{-1} (C=O ester), C–H absorption band at 1363 cm^{-1} (–/C–/CH3), and –C–/O–/ stretching band at ~1260 cm^{-1} confirmed the formation of ester bonds. It is also evidenced an increase in the intensity of OH in plane bending vibration at 1363 cm^{-1}, band specific to the wood polysaccharides components, cellulose and hemicelluloses.

X-ray Photoelectron Spectroscopy Analysis (XPS)

The analysis of the survey spectra indicates the presence of carbon, oxygen and reduced amounts of nitrogen, these being the expected elements in softwood species. The surface elemental compositions of wood samples, non-treated and treated with SA, are summarized in Table 1.

Table 1

Surface elemental composition of untreated and treated wood analyzed by XPS

Sample	C %	O %	N %	O/C	N/C
R	67.46	29.64	2.27	0.4394	0.0336
MW(SA60)	62.79	35.67	1.28	0.5681	0.0204
MW(SA80)	63.46	34.72	1.71	0.5471	0.0269
MW(SA120)	64.50	34.00	1.21	0.5271	0.0187

The O/C ratio (as indication of surface oxidation) and distribution of carbon and oxygen atoms of chemical treated softwood samples differ considerably from those obtained for non-modified wood. As it can be observed from Fig. 6-9, the intensity for high resolution C_{1s} peaks decreased for chemically modified wood samples comparatively with reference samples due to the chemical reaction between SA and wood polymer constituents. C_{1s} peak is of greatest importance when discussing XPS data recorded for wood samples. Deconvolution of carbon and oxygen peaks was performed and their assignment is presented in Table 2.

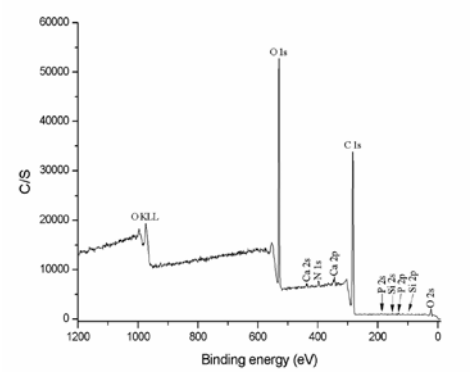

Fig. 6.
XPS spectrum for reference (R sample).

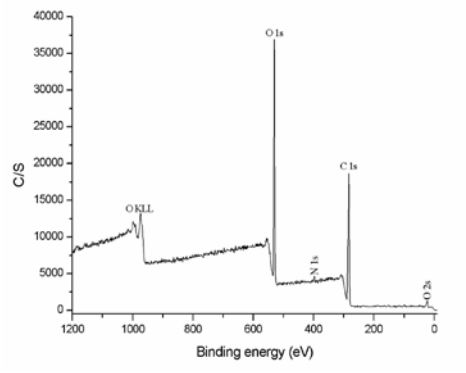

Fig. 7.
XPS spectrum for MW(SA60) sample.

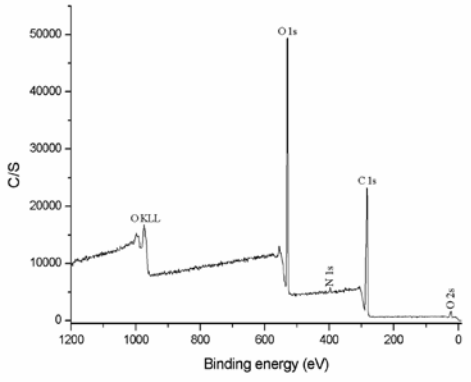

Fig. 8.
XPS spectrum for MW(SA80) sample.

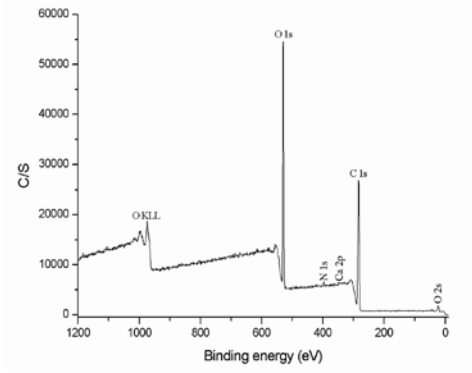

Fig. 9.
XPS spectrum for MW(SA120) sample.

Table 2

The assignment of deconvoluted carbon and oxygen peaks

Symbol	Carbon atom or oxygen atom bound to
C1	C–C or/and C–H
C2	C–O
C3	C=O or/and O–C–O
C4	O–C=O
C5	$CO\text{-}CH_3$ or/and $CO\text{-}CH_2$
O1	O–C=O
O2	C–O or/and C-O-C
O3	O=C or/and O-C-O

From the XPS data of the non-modified and modified softwood samples the carbon peaks components (C_{1s}) as well the oxygen peaks components (O_{1s}) were calculated and given in Table 3.

Table 3

Summary of XPS spectral parameters in softwood

Sample	Parameter	C_{1s}					O_{1s}		
		C_1	C_2	C_3	C_4	C_5	O_1	O_2	O_3
R	BE (eV)	285.00	286.50	287.90	289.2	-	533.5	532.90	531.6
	RC (%)	43.12	40.43	11.95	4.5	-	24.04	65.06	10.90
MW(SA60)	BE (eV)	285.00	286.70	288.20	289.2	285.60	533.6	532.80	532.1
	RC (%)	28.49	35.24	6.43	16.99	12.85	24.03	65.05	10.92
MW(SA80)	BE (eV)	285.00	286.70	288.10	289.3	285.5	533.5	532.80	532.1
	RC (%)	23.54	33.10	6.87	19.65	16.84	34.38	45.18	20.44
MW(SA120)	BE (eV)	285.00	286.70	287.90	289.3	285.50	533.5	532.80	532.1
	RC (%)	23.87	31.97	5.56	19.09	19.51	39.16	37.20	23.64

where: BE- Binding Energy; RC- Relative Concentration

For chemically modified wood, the highest amount from carbon peaks components was found for C_2 class consisting of C-O linkage of carbon (32-35%) due the reagent used for chemical modification. The C4 class consisting of O-C=O linkage of carbon present a significant increase for treated wood comparatively with reference. The C5 class representing the $CO\text{-}CH_3$ and/or $CO\text{-}CH_2$ groups contributed with 13-19% for modified wood samples depending of SA concentration.

Thermal Analysis (TGA)

In thermal analysis of wood samples, a common behavior is the dehydration process in which 5-8% of adsorbed water is removed, with no degradation process up to 160^0C. Above this temperature the thermal stability gradually decreases and decomposition takes place. It is well known that the structural organization of polymers influences the shape of thermograms in thermal analysis of wood materials. Wood samples are known to present different degradation profiles depending on the wood chemical composition. Cellulose is highly crystalline, which makes it thermally stable. Hemicelluloses and lignin, on the other hand, are amorphous and start to degrade before cellulose. Hemicelluloses are the least thermally stable wood components, due to the presence of acetyl groups. Thermograms recorded for the wood samples, non-modified and modified with SA are shown in Figs. 10-13. For all wood samples under study, there are noticed two main degradation processes.

The chemical modification of wood by reaction with succinic anhydride decreases thermal stability of wood which presents a lower temperature of decomposition and a higher weight loss rate than that of reference wood samples. The mass losses, at around 100°C, are related to water evaporation. TG analysis of reference wood samples indicated a loss of water of 5.1% between 20°C and 140°C. A loss of 3.03 - 3.12% of water is observed over the same temperature range for modified wood samples. Thermal parameters presented in Table 2 evidence a decrease of thermal stability through chemical modification of wood using treatment with succinic anhydride in solvent.

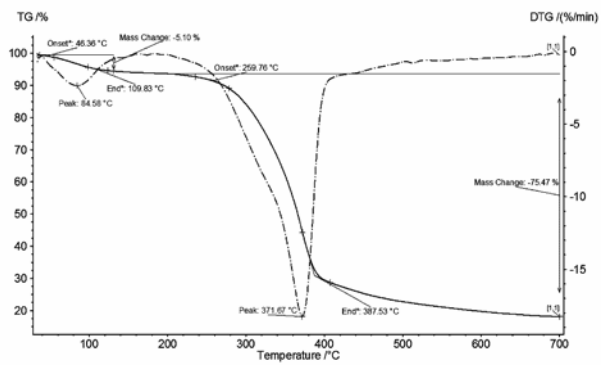

Fig. 10.
TG-DTG curves for reference sample.

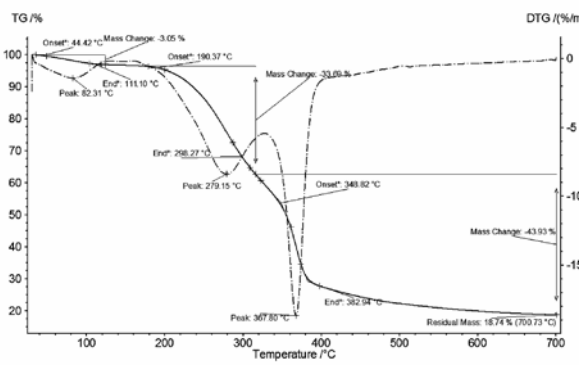

Fig. 11.
TG-DTG curves for MW(SA60) sample.

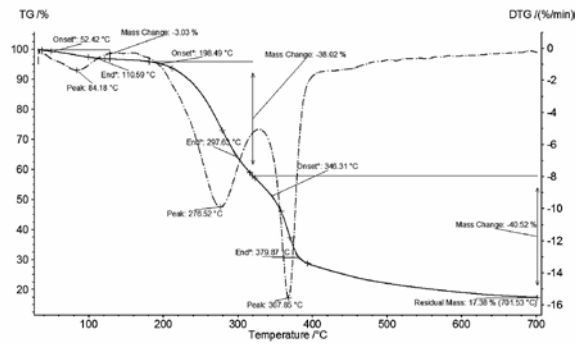

Fig. 12.
TG-DTG curves for MW(SA80) sample.

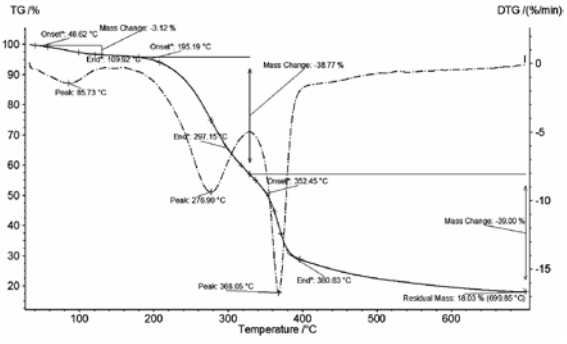

Fig. 13.
TG-DTG curves for MW(SA120) sample.

Table 2

TG-DTG parameters recorded for modified wood samples

Sample	T_i °C	Peak °C	T_f °C	Weight loss %	T_i °C	Peak °C	T_f °C	Weight loss %	Residual mass %	T_5 °C
R	46.3	84.6	109.8	5.10	259.8	371.7	387.5	75.5	18.11	111.5
MW(SA60)	44.4	82.3	111.1	3.05	348.8	367.8	382.8	43.9	18.74	202.8
	190.37	279.3	298.27	33.69				77.6		
MW(SA80)	52.4	84.2	110.6	3.03	346.3	367.9	379.9	40.5	17.38	197.7
	198.5	276.5	297.6	38.02				78.5		
MW(SA120)	48.6	85.7	190.9	3.12	352.5	368.1	380.8	39.0	18.03	195.5
	195.2	276.9	297.2	38.77				77.8		

The temperature corresponding to 5% weight loss (T_5) exhibits an increase for chemically modified wood by comparison with the reference samples. The active decomposition temperatures that caused the major weight loss were 371.7°C for reference wood sample, 367.8°C for MW(SA60) sample, 367.9°C for MW(SA80) sample, and 368.1°C for MW(SA120) sample, respectively.

CONCLUSIONS

The chemical treatment with organic anhydride induced structural changes in wood samples. The spectral changes registered through FTIR and XPS spectroscopy methods confirmed the chemical modification of wood by reaction with succinic anhydride. A stronger carbonyl band at 1725 cm^{-1} is observed for chemically modified wood with increasing SA concentration. This enhanced carbonyl absorption peak (C=O ester), as well as the C–H absorption band at 1363 cm^{-1} (–/C–/CH3), and –C–/O–/ stretching band at ~1260 cm^{-1} confirmed the formation of ester bonds. Results obtained from XPS analysis indicate an increase in the O/C ratio values for chemically modified wood due to the oxidation processes occurred after treatment. The thermal decomposition of wood (non-modified and modified) is a complex process because the wood chemical structure is very complex with polymer components which degrade differently. The chemical modification of wood by reaction with succinic anhydride decreases thermal stability of wood which presents a lower temperature of decomposition and a higher weight loss rate than that of reference wood samples.

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ENHANCING THE ABRASION RESISTANCE OF PMMA/ATH LAYERS REALISED BY MEANS OF ATMOSPHERIC PRESSURE PLASMA POWDER DEPOSITION ON WOOD

Lena WALLENHORST
University of Applied Sciences and Arts, Laboratory of Laser and Plasma Technologies
Von-Ossietzky-Straße 99, 37085 Göttingen, Germany
E-mail: lena.wallenhorst@hawk-hhg.de

Roman RERICH
University of Applied Sciences and Arts, Laboratory of Laser and Plasma Technologies
Von-Ossietzky-Straße 99, 37085 Göttingen, Germany
E-mail: roman.rerich@hawk-hhg.de

Matej VOVK
University of Ljubljana, Biotechnical Faculty
Jamnikarjeva 101, 1000 Ljubljana, Slovenia
E-mail: matejvovk@yahoo.com

Sebastian DAHLE
Clausthal University of Technology, Institute of Energy Research and Physical Technologies
Leibnizstraße 4, 38678 Clausthal-Zellerfeld, Germany
E-mail: s.dahle@pe.tu-clausthal.de

Holger MILITZ
Wood Biology and Wood Products, Burckhardt Institute, Georg-August-University Göttingen
Büsgenweg 4, 37077 Göttingen
E-mail: hmilitz@gwdg.de

Gisela OHMS
University of Applied Sciences and Arts, Laboratory of Analytical Measurement Techniques
Von-Ossietzky-Straße 99, 37085 Göttingen, Germany
E-mail: Gisela.ohms@hawk-hhg.de

Wolfgang VIÖL
University of Applied Sciences and Arts, Laboratory of Laser and Plasma Technologies
Fraunhofer Institute for Surface Engineering and Thin Films, Appl. Center for Plasma and Photonics
Von-Ossietzky-Straße 100, 37085 Göttingen, Germany
E-mail: wolfgang.vioel@hawk-hhg.de

Abstract

In this article, we present the deposition and stabilisation of PMMA/ATH powder on wood and glass substrates in a cold atmospheric pressure plasma-spray process. For this purpose, the raw powder material was injected to the effluent plasma of a jet discharge to coat the substrate that is situated in a comparatively cold region of the afterglow. Since the abrasion resistance of pure PMMA/ATH powder coatings deposited with the use of compressed air as process and carrier gas turns out to be very low, two variations to stabilise those coatings were tested. The replacement of compressed air as process and carrier gas by forming gas did not lead to an increased abrasion resistance, but the addition of phenol-formaldehyde (PF) powder successfully stabilised the coatings.

Key words: *plasma coatings; PMMA/ATH; abrasion; recycling.*

INTRODUCTION

Poly(methyl methacrylate) (PMMA) reinforced with aluminium trihydrate ($Al(OH)_3$, ATH) is classified as solid surface material (ISO 19712-2, 2007) with characteristics meeting the requirements of the performance standard ANSI/ICPA SS-1-2001. The PMMA/ATH composite, as it is abbreviated, was developed and described by Duggins and co-workers (Duggins and Ford 1974; Duggins et al. 1974). In general, the composites contain about 50 to 70 wt.% of ATH filler and about 30 to 50 wt.% of

PMMA resin. Because of this composition, machining of PMMA/ATH composite is very similar to machining of wood, but PMMA/ATH can additionally be thermoformed similar to thermoplastics. Unlike wood, though, it exhibits a high moisture resistance along with both a good chemical and UV resistance. These properties make PMMA/ATH composites widely useable as working surfaces and claddings for indoor and outdoor applications.

However, about 20% of the material produced is represented in post-industrial waste, leading to a production of about 1000 tons of PMMA/ATH waste every year only in Slovenia. Cut-offs and powder waste of PMMA/ATH material is generated during the post-polymerization process by trimming and sanding the PMMA/ATH sheets. Different methods of recycling that particular waste have already been considered (Kaminsky and Franck 1991; Kaminsky et al. 2004; Hochberg and Young 1988; Šušteršič et al. 2013; Wallenhorst et al. 2015; Tušar et al. 2014; Tušar et al. 2015), but none of them have provided a simple, sustainable and cost-effective solution yet. In previous studies (Wallenhorst et al. 2015), PMMA/ATH powder was deposited in a plasma-spray process by the use of air as process and carrier gas. A functionalisation of the used PMMA/ATH material could be detected; however, it did not yield abrasion resistant coatings. Therefore, the use as protective coatings on wood was not possible. In the present study, the replacement of air by forming gas was investigated which could yield more reactive molecule fragments and consequently improve the adhesion.

Our preliminary research suggested that PMMA/ATH boards and waste material are compatible with phenol formaldehyde (PF) resin, yielding the opportunity to use this resin in a production of some kind of recycled composite. PF resins are most often used in wood composite adhesives (United states department of agriculture 2010). It has a unique ability to react with formaldehyde in acidic or basic conditions (Pilato 2010). The former gives resole resins, while the reaction in basic conditions yields novolak resins (Mittal and Pizzi 2003). Powdered novolak PF can easily be combined with the PMMA/ATH powder for the application by atmospheric pressure plasma powder deposition. The use of powdered phenol novolak resin with a medium hexamethylenetetramine (HMTA) content and medium flow turned out to be appropriate for the intended application. This kind of resin was intentionally formulated as a binding agent for the manufacturing of hot or highly compressed cold pressed grinding wheels, where high thermal resistance and toughness is required. The performance of powdered phenolic resin is roughly determined by its molecular weight, which influences flow, hexamine content and particle size (Pilato 2010). The resin used in this study melts at about 90 °C and reacts at 130°C. When cured, it has high dry and wet strength and is very resistant to water and damp atmospheres (United states department of agriculture 2010).

OBJECTIVES

The main objective of this study was the stabilisation of plasma coatings based on PMMA/ATH powder as protective coating on wood from a recycled waste powder.

METHODS, MATERIAL AND EQUIPMENT

Atmospheric pressure plasma layer deposition

To deposit composite PMMA/ATH layers, a setup as described in (Gascón-Garrido et al. 2016a, 2016b; Wallenhorst et al. 2015) was used which is based on the Plasmabrush from Reinhausen Plasma GmbH with a modified spraying nozzle. Briefly, the powder was added to the afterglow of a jet discharge driven by pulsed high voltage (ignition voltage: approx. 15 kV, effective voltage: 2-3 kV, max. input power: 2 kW, pulse duration: 5-10 µs, pulse repetition rate: 50 kHz). To generate the particle aerosol, the dry powder was homogeneously compressed in a cylindrical container and moved towards a rotating brush at a constant velocity defining the feed rate. Above the brush, the carrier gas stream took up the particles. The PMMA/ATH powder was sieved prior to utilisation, yielding initial diameters below 70 µm. The samples were placed on an x-y linear stage that moved them below the spraying nozzle to ensure an overall homogeneous coating.
Further, in an attempt to improve the abrasion resistance, two different modifications of this basic deposition process were investigated:

A. Use of pure PMMA/ATH powder with compressed air as process and carrier gas,
B. Use of forming gas instead of compressed air as process and carrier gas,
C. Addition of 10 % phenol-formaldehyde powder (PF, Borofen BL-35 from Kolpa d.d) to the PMMA/ATH powder.

The coatings were deposited on beech wood (Fagus sylvatica L., 4 x 76 x 26 mm³) as well as on soda-lime glass (microscope slides) as a model substrate for surfaces that cannot be coated easily. The parameters applied during the deposition process are listed in Table 1; for each coating system, two replicates were prepared. Since every substance that was deposited in this plasma process requires different parameters to ensure homogenous coatings and to keep the substrates' temperatures low enough to avoid thermal damage at the same time, the deposition parameters cannot be kept the same for all coating systems.

Table 1

Summary of coating parameters (subscript w stands for wood, g for glass).

Sample No.	Deposited material	Process/carrier gas	Carrier gas pressure in bar	Working distance in mm	Powder feed rate in cm³/h	Substrate displacement in mm/s
$A_{w/g}$	PMMA/ATH	Compressed air (40 l/min)	1.5	13	90	20
$B_{w/g}$	PMMA/ATH	Forming gas (35 l/min)	1.0	25	150	100
$C_{w/g}$	PMMA/ATH (90 %) PF (10 %)	Compressed air (40 l/min)	1.6	16	150	20

Abrasion tests

To study the abrasion resistance, a setup according to DIN ISO 9211-4 was used. Here, cotton tissues were fixed on a test probe and moved on the sample with a pressure of (5±1) N. One cycle is defined as one movement backwards followed by one movement forwards. The samples were evaluated visually after taking a picture with an EOS 600D digital camera (Canon Inc.).

RESULTS AND DISCUSSION

Figure 1 shows pictures of the three different coating systems on wood after one abrasion cycle. Except for some particles that remained fixed in the wood's rough surface, the two coatings A_w and B_w were already removed, hence, exchanging compressed air as process/carrier gas by forming gas does not improve the abrasion resistance. In contrast, only slight changes could be observed for the coating consisting of PMMA/ATH and PF. Similarly, coating systems A_g and B_g on glass substrates (not shown) were completely removed after 1 cycle whereas system C_g showed only little removal of substance.

For the coating system containing PMMA/ATH and PF, the abrasion tests were extended. Figure 2 shows coated glass and coated wood surface after subjection to 1000 abrasion cycles, still with a significant amount of coating on the ablation site. For wood, the coating could be removed after approximately 2000 cycles, whereas no complete removal of the coating on glass was possible (up to 4400 cycles were tested on each sample). Hence, both, the adhesion between the particles that constitute the coating as well as the adhesion between the coating and the surfaces, were improved.
The poorer abrasion resistance in the case of wood substrates might be explained by the lower mechanical stability of wood which could lead to wood failure instead of a failure of the adhesive bond. Laborie and Frazier (Laborie and Frazier 2006) proposed secondary interactions between the wood constituents and the hydroxyl groups of the resin as mechanism explaining the adhesion between wood and PF.

(a) (b) (c)

Fig. 1.
Coating after 1 cycle. (a): sample A_w (PMMA/ATH with compressed air); (b): sample B_w (PMMA/ATH with forming gas); (c) sample C_w (PMMA/ATH + PF with compressed air).

(a) (b)

Fig. 2.
PMMA/ATH + PF coating after 1000 cycles on glass (Fig. 2a) and wood (Fig. 2b).

CONCLUSIONS

In this article, a way to deposit abrasion-resistant coatings on wood and glass via the addition of phenol-formaldehyde powder to the PMMA/ATH raw powder was presented. The PMMA/ATH + PF coatings withstand approx. 2000 abrasion cycles on wood, whereas it was not even possible to completely remove the coatings from glass substrates by the method applied. To understand the mechanisms behind this significantly improved abrasion resistance, detailed studies on the chemical and structural properties are subject to further ongoing investigations.

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PROPERTIES OF WOOD-STRAW COMPOSITES BONDED WITH MODIFIED UF ADHESIVE AND PRE-TREATED STRAW PARTICLES

Pavlo BEKHTA
Ukrainian National Forestry University,
Department of Wood-Based Composites, Cellulose and Paper
Gen.Chuprynky 103, 79057, Lviv, Ukraine
E-mail: bekhta@nltu.edu.ua

Emilia-Adela SALCA
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: emilia.salca@unitbv.ro

Ruslan KOZAK
Ukrainian National Forestry University,
Department of Wood-Based Composites, Cellulose and Paper
Gen.Chuprynky 103, 79057, Lviv, Ukraine
E-mail: kozak@nltu.edu.ua

Abstract

The paper presents the effect of some non-expensive treatments applied before the particleboards production under two instances, in one case by adding ethanol to the UF resin and in other cases by boiling the straw particles, with a view to improve the bonding quality of the mixed wood-straw composites. The modulus of rupture (MOR) and tensile strength perpendicular to the surface (IB) were evaluated. Mechanical performance of wood-straw particleboards bonded with the UF modified glue by ethanol and that one of the particleboards manufactured from pre-treated straw raw material by boiling in soapy solution met the standard requirements. The results of this study show the potential of such agro-wastes for the particleboards manufacturing.

Key words: *wood-straw particleboards; wheat straw; wood particles; urea formaldehyde.*

INTRODUCTION

Particleboard is one of the most used materials for constructions, furniture, cabinetry, walls, flooring, and other aesthetic architectural applications. A great interest was shown the last decades to sustainable materials, approach that brings to light the use of various wastes, such as vegetable agro-waste to produce new materials. Therefore the use of conventional wooden material for particleboards has shifted to the use of renewable resources (Cuk et al. 2011). Even there have been few limitations connected to the lack of chemical knowledge and properties of these agro-materials, over the years wood chips have been substituted in particleboards production by particles obtained from annual plants, such as: wheat straw, rice straw, tea leaves, coconut chips, flax, hemp, kenaf, bamboo, bagasse, almond shell, corn peel, sunflower, sugar cane, and rapeseed, just to name a few (Boquillon et al. 2004; Dukarska et al. 2016; Guler et al. 2016; Guru et al. 2006; Han et al. 1998; Kalaycioglu and Nemli 2006; Li et al. 2010; Papadopoulos and Hague 2003; Xu et al. 2004; Yalinkilic et al. 1998).

According to FAO, the world cereal production went along with its utilization and stocks reaching more than 2400 million tons in 2016, out of which a total of 760.1 million tons was recorded for wheat production. Wheat straw is still one of the most abundant and cheap agro-waste materials in the world. The wheat straw is currently used in some limited applications: feed stuff, fertilizer, pulp industry, nano-materials, and for bio-ethanol also in pyrolysis, combustion and gasification (Talebnia et al. 2010). In Ukraine the wheat straw is considered by far the most perspective raw material suitable and very attractive to be used for particleboards manufacturing (Bekhta 2007).

Ukraine has been rated in 2011 among the top ten countries of wheat production and consumption per capita, and exports as well (FAO). According to The State Statistics Committee in 2015 the grain production in Ukraine was the third highest ever. In general, previous three years were the most productive throughout the history of Ukraine. Also the forecast for the crop production in 2016/17 was estimated at 24 million tons due to the favorable spring weather that has greatly improved the wheat yield prospects. Nowadays straw and other agricultural residues represent the most important sources of biomass for energy in Ukraine.

Since 2010 straw has been used for the production of pellets and briquettes (Geletukha et al. 2015). There are big amounts of wheat straw residues which are still burnt in the field causing significant environmental problems apart the loss of a valuable resource (Bekhta et al. 2013).

Wheat straw fiber and wood present different morphological features and mechanical properties, and in general they have a similar chemical composition, containing cellulose, hemicellulose, lignin and some extractives. The high content of silica in wheat straw leads to greater power consumption and also limits the service life of the crushing equipment. The fat-wax surface layer worsens wetting and gluing and it influences the adhesion between particles and represents a major obstacle for the particleboards production.

The quality of bonding may be improved when removing the fat-wax layer by using some physical and chemical processes (Bekhta et al. 2011) or with glues having greater reactivity instead of urea formaldehyde (UF) glue (Pease 1998; Grigoriou 2000; Zhang et al. 2011). Although it has some disadvantages, such as formaldehyde emissions it appeared that UF is still the most economical, due to its low cost and easy production, although it produces a low bonding with the straw particles (Guru et al. 2006). But the bonding quality of wheat straw with UF resin may be improved by applying several treatments either to the raw material or to the adhesive itself (Bekhta and Kozak 2011; Bekhta et al. 2013).

OBJECTIVE

The paper presents the effect of some non-expensive treatments applied before the particleboards production under two instances, in one case to the UF resin by adding ethanol and in another case to the straw particles by boiling them, with a view to improve the bonding quality of the mixed wood-straw composites. The modulus of rupture (MOR) and tensile strength perpendicular to the surface (IB) were evaluated.

MATERIAL, METHOD, EQUIPMENT

Wood particles supplied by a particleboards producer and wheat straw resulted from a local farm in Ukraine were used to produce experimental particleboards. Stems of wheat straw were cut, crushed and dried at 4% MC while wood chips were used as they were supplied and dried separately at same moisture content. A commercial UF resin with a solid content of 65% and ammonium chloride as a hardener were used for the particleboards manufacturing. The same pressing schedule of the wood-straw particleboards was applied, such as: the temperature of 170°C and the pressure of 2.2 MPa for 6 min. The panels (300 x 300 mm) were produced having the same target density of 650 kg/m^3. Panels with no treatment were manufactured as control panels. The study cases as function of the pre-treatment applied before the particleboards manufacturing, in one case to the UF resin and in the other cases to the wheat raw material, are presented in Table 1. The experimental work was performed at the Department of Wood-Based Composites, Cellulose and Paper from the Ukrainian National Forestry University in Lviv, Ukraine.

Table 1

Schedule of the experimental work

Case and Treatment type	**Case A**	**Case B**	**Case C**
	UF resin modified by ethanol	**Pre-treatment applied to straw by boiling**	
Type of board and thickness	three-layered panel 16 mm	single-layer panel 19 mm	
Ratio between layers	20:60:20 (outer:inner:outer)	60% wood particles and 40% wheat straw	
Treatment	outer layers: wood particles + UF glue inner layer: wood and straw particles + modified UF glue (10 mass units of ethanol on 100 mass units of resin)	45 min- boiling in soapy solution of 20% concentration	45 min - boiling in water at 100°C

Prior to sampling the manufactured particleboards were kept in laboratory conditions at the temperature of 20ºC and 65% RH to reach the equilibrium moisture content. The modulus of rupture

(MOR) and the tensile strength perpendicular to the surface (IB) were determined according to standards (BS EN 310 1993; BS EN 319 1993).

RESULTS AND DISCUSSION

The results for the mechanical properties (MOR and IB) obtained for all the cases under study are graphically displayed in Figures 1 and 2.

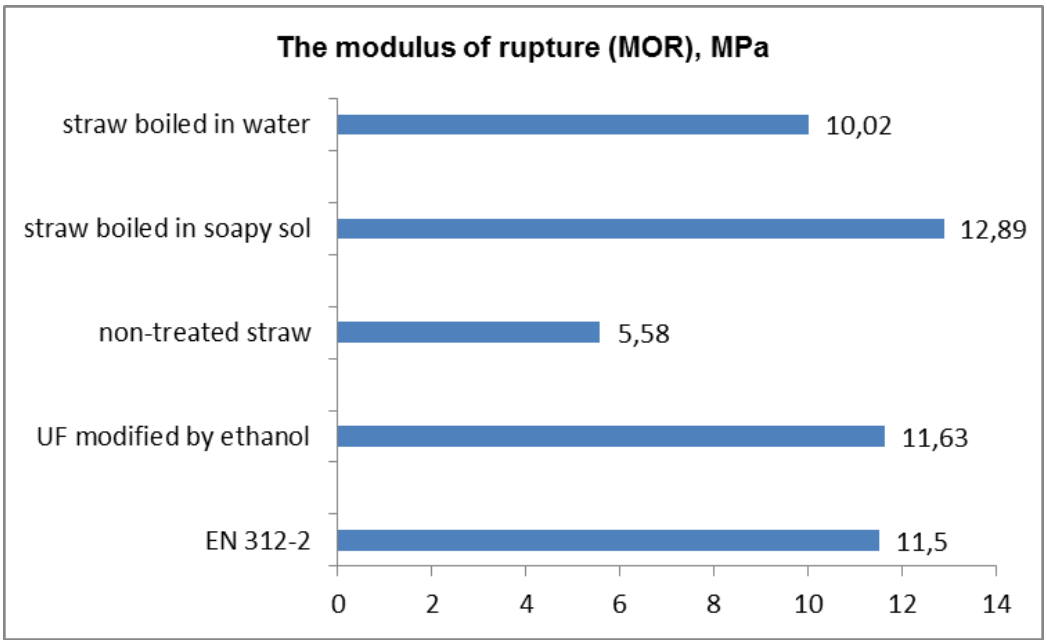

Fig. 1.
Modulus of rupture of wood-straw particleboards as function of different treatment applied before manufacturing.

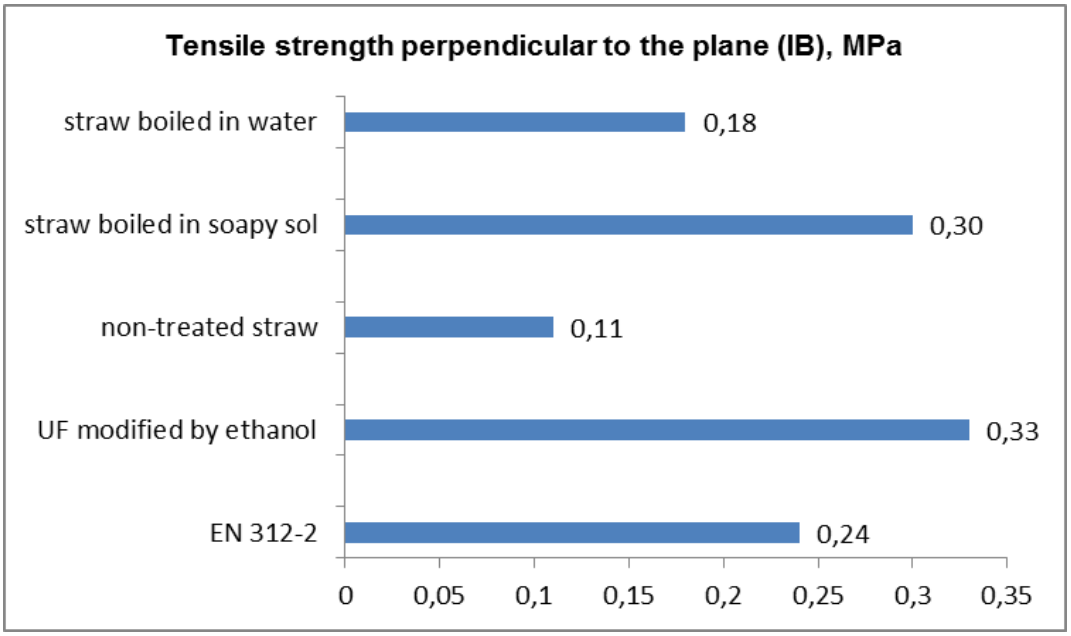

Fig. 2.
Tensile strength perpendicular to the surface of wood-straw particleboards as function of different treatment applied before manufacturing.

Case A - UF resin modified by ethanol

The results show that the wood-straw composites glued with modified UF resin by ethanol exhibited better properties when compared to those requested by standards specific to wood particleboards (EN 312 2003). The ethanol contained by the glue was expected to dissolve the fat-wax layer of straw during the particleboards manufacturing. Therefore it was assumed that the hydrophobic effect of fat-wax layer on the adhesive interaction with particles decreases. During the pressing step the ethanol evaporates from the boards together with the moisture (Kozak et al. 2016).

Cases B and C - Pre-treatment applied to straw by boiling (soapy solution and water)

Both types of mixed particleboards made of wheat straw particles boiled in a soapy solution and water showed increased MOR and IB values when compared to that of control samples. The treatments applied to the raw material improved the properties of the samples and such result may be related to the dissolution of silica in the raw material when being exposed in such conditions. The reduction of silica in the straw particles may positively influence the bonding quality and the distribution of glue on the particles. When compared to the requirements of EN standards (EN 312 2003) it appeared that only the composite panels made of wheat straw particles pre-treated by boiling in soapy solution met them.

Neither the control panels nor the boards made of boiled straw particles met those standard requirements. Therefore the pretreatment of straw particles with a soapy solution was found to be the most effective way to improve the mechanical properties of the wood-straw composites, such as the internal bond strength of the samples. Such result may be explained by the improved wettability of the raw material surface and consequently the subsequent improvement in adherence of UF resin and the hydroxyl groups of cellulose. The surface-active agents in the soapy solution contributed to the strong effect of the solution on the internal bonds in particleboards (Bekhta et al. 2013).

Similar results and conclusions from this work on the potential of such wood-agro-wastes particleboards were found by other authors in the field of composites (Boquillon et al. 2004; Mo et al. 2003; Zhang et al. 2011).

CONCLUSIONS

Mechanical performance of wood-straw particleboards bonded with the UF modified glue by ethanol and that one of the particleboards manufactured from pre-treated straw raw material by boiling in soapy solution met the standard requirements specific to particleboards.

The results of this study show the potential of agro-wastes for the particleboards manufacturing.

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IMPROVING THE DURABILITY OF COMMON HORNBEAM WOOD (*Carpinus betulus* L.) ACETYLATED WITH ACCOYA® METHOD

Fanni FODOR
University of Sopron, Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts
Bajcsy-Zs. Str. 4, 9400 Sopron, Hungary
Tel: 0036 99 518 399, Fax: 0036 518 647, E-mail: fodor.fanni@phd.uni-sopron.hu

Chiel LANKVELD
Accsys Technologies PLC Group
Westervoortsedijk 73, 6827 AV Arnhem, Netherlands
E-mail: chiel.Lankveld@accsysplc.com

Róbert NÉMETH
University of Sopron, Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts
Bajcsy-Zs. Str. 4, 9400 Sopron, Hungary
E-mail: nemeth.robert@uni-sopron.hu

Abstract

The main objective of the present research was to examine the effect of acetylation on the durability of common hornbeam wood (Carpinus betulus L.). For this, a short-term (16-week-long) fungi culture test and long-term field test was carried out.

The laboratory tests were done according to EN 113 with three different fungi: Coniophora puteana and Poria placenta brown-rot fungi and Coriolus versicolor white-rot fungus. The durability class was determined according to mass loss after 16 weeks.

The field test is carried out according to EN 252 in ground contact. The durability of hornbeam, acetylated hornbeam, beech and pine sapwood is evaluated and the durability class is determined in every 6 months from 2016 July.

According to the results, the durability classes were determined for each species and samples, and conclusions were made on the effect of acetylation.

Key words: *fungi resistance; hornbeam; acetylation; soil; durability.*

INTRODUCTION

Over the past decades the use of renewable raw materials increased. This attitude is also reflected in the case of wood modification which is used to improve the material properties, but produces a non-toxic material which can be disposed of at the end of a product lifecycle without presenting an environmental hazard any greater than unmodified wood. The use becomes more widespread each year. This is also due to new restricting laws and regulations concerning wood preservatives.

Acetylation is a chemical modification process that has been studied by scientists around the world for almost 90 years (Fuchs 1928). A pilot plant was built in the Netherlands in 2004 and after 3 years a large scale commercial facility was constructed in 2007 and the production of acetylated wood began. Acetylated wood is now specified and used globally in residential and commercial applications.

During acetylation acetic anhydride is used as a dehydrating agent. The hydroxyl groups are replaced by acetyl groups which results in a dimensionally stable wood. There are many characteristics of wood (like extractives, sample parameters, moisture, density, permeability and wood quality) and treatment settings (like catalysts, purity of the anhydride, initial moisture content, temperature, pressure, etc.) that influence the final products' properties. After acetylation the samples are taken for chemical quality assurance and the weight percentage gain is calculated (WPG).

The acetylated material is much more resistant against any biological attack. It is the WPG rather than OH substitution that determines the degree of decay resistance (Hill *et al.* 2003). Many studies have been performed using laboratory pure culture tests, sterile and unsterile laboratory soil burial tests, and in long-term outdoor exposure tests which showed the optimal WPG above which no microbial degradation (usually weight loss) of the wood occurs. It cannot be extrapolated which WPG is sufficient against any biological attack in case of any wood species, but it can be concluded that the higher the level of acetylation is the higher the durability will be.

Multiple scientific papers have proven a direct link between acetyl content and wood durability (Goldstein *et al.* 1961, Peterson and Thomas 1978, Imamura and Nishimoto 1987, Takahashi *et al.* 1989, Beckers *et al.* 1994, 1995, Larsson and Simonson 1999, Ohkoshi *et al.* 1999, Suttie *et al.* 1999, Larsson *et al.* 2000, Papadopoulos and Hill 2002, Hill *et al.* 2006) but there are also other factors which influence like water uptake, density, anatomy, etc. According to Militz (1991) beech resisted brown and white rot fungi at 20% WPG. It needs 12% WPG against *Coriolus versicolor*, 20% WPG against *Poria placenta*, 17% WPG against *Coniophora puteana* and *Gloeophyllum trabeum,* and 10% WPG against soft rot decay (Beckers *et al.* 1994). In field tests acetylated beech had better fungal resistance than natural beech however even at the highest WPGs (18-20%) the fungi were at least in their developing stages to attack the acetylated wood (Mohebby and Militz 2010).

OBJECTIVE

The main objective of the present research was to examine the effect of acetylation on the durability of hornbeam wood. For this, a short-term (16-week-long) fungi culture test and long-term field test was carried out.

MATERIAL, METHOD, EQUIPMENT

Edged and air-dry hornbeam boards were ordered from a Hungarian sawmill (BOPAÁR Ltd.). The dimensions were 27 × 160 × 2500mm (thickness × width × length). Half of the boards were left untreated and the other half was sent to Accsys Technologies to be acetylated under industrial conditions. The average WPG was 15%.

Short-term fungi culture test

The fungi resistance of natural and acetylated hornbeam was determined according to EN 113:1996, which defines it by the loss in mass of the specimens in percentage after 16 weeks of exposing them to fungi. The culture medium was Merck malt extract agar 1.05398.0500.

There were three fungi used according to the standard:

- *Coniophora puteana* brown-rot fungus
- *Poria placenta* brown-rot fungus
- *Coriolus versicolor* white-rot fungus

The sample dimensions were 15 × 25 × 50 mm (thickness × width × length) at 12 % moisture content. There were 2-4 virulence vessels with only untreated samples: these indicated the intensity of the fungal attack and determined the mass loss of unmodified wood in the decay experiment. There were 11 reference vessels which contained one untreated and one acetylated hornbeam sample – here the reagent can have influence on the mass loss of the untreated sample.

After 16 weeks the samples were taken from the vessels, cleaned and dried to constant mass. Then the mass loss was calculated and the durability class was determined according to standard: not durable (>30%), slightly durable (>15%), moderately durable (>10%), durable (>5%) and very durable (≤5%).

Long-term field test

The durability in ground contact was tested according to DIN EN 252:2014 with some slight changes. The sample dimensions were changed to 20 × 50 × 300 mm (thickness × width × length). There were twelve stakes of each type: hornbeam, acetylated hornbeam, supplemented with beech and pine sapwood according to standard. The beech and pine stakes indicate the intensity of the decaying mechanism of the soil. The stakes were put to exposure in the Outdoor Exposure Testing Field of the Department of Wood Science in Sopron in 2016 April. The durability class of each stake was evaluated every 6 months according to standard.

RESULTS AND DISCUSSION

Short-term fungi culture test

Acetylation improved the fungi resistance of hornbeam to a great extent as it seen in Table 1. It lost less than 1% of weight when it was exposed to three different fungi. When the untreated specimen was accompanied by an acetylated specimen, the untreated one lost less weight than the virulence samples because of the reagent's presence in the flask. In some cases, the fungi could not degrade the wood material because of the appearance of mould, these were excluded from the results. In case of *Poria placenta*, 4 out of 9 acetylated samples showed negative weight loss which were changed to zero according to standard.

Coniophora puteana attacks both softwood and hardwood but has a preference for conifers. In the case of virulence samples, the fungi successfully decayed the material, but when the samples were mixed, the reference controls were more prone to be attacked by mould (Fig.1.).

Poria placenta attacks primarily conifers but the weight loss values (20.78%) indicate that the test was successful (in case of conifers it would have been around 35-40%). The mould is present in many instances also and the deviation is larger than in the case of the other two fungi (Fig.2.).

Coriolus versicolor attacks primarily broadleaved species, which explains the small deviation and the scarce appearance of mould (which could not hinder the decaying process). In this case, there is only small difference between the virulence and reference untreated samples (Fig.3.).

Table 1

Weight loss of untreated and acetylated samples exposed to wood-decay fungi for 16 weeks. (SD): standard deviation.

Weight loss (%)			Average	SD	Durability class
Coniophora puteana	Virulence	Control	45.64	3.68	Not durable (1)
	Reference	Control	18.58	1.07	Slightly durable (2)
		Acetylated	0.84	0.17	Very durable (5)
Poria placenta	Virulence	Control	20.78	1.54	Slightly durable (2)
	Reference	Control	21.19	6.61	Slightly durable (2)
		Acetylated	0.20	0.21	Very durable (5)
Coriolus versicolor	Virulence	Control	34.00	1.91	Not durable (1)
	Reference	Control	32.66	2.09	Not durable (1)
		Acetylated	0.83	0.12	Very durable (5)

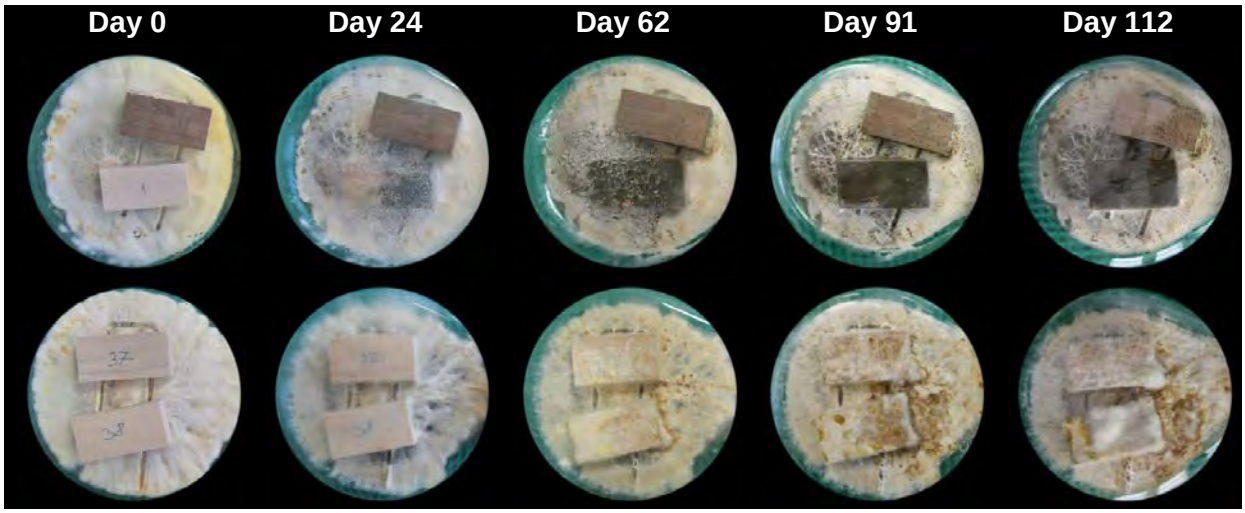

Fig. 1.
Photo series of reference (above) and virulence (below) samples' fungi resistance (Coniophora puteana).

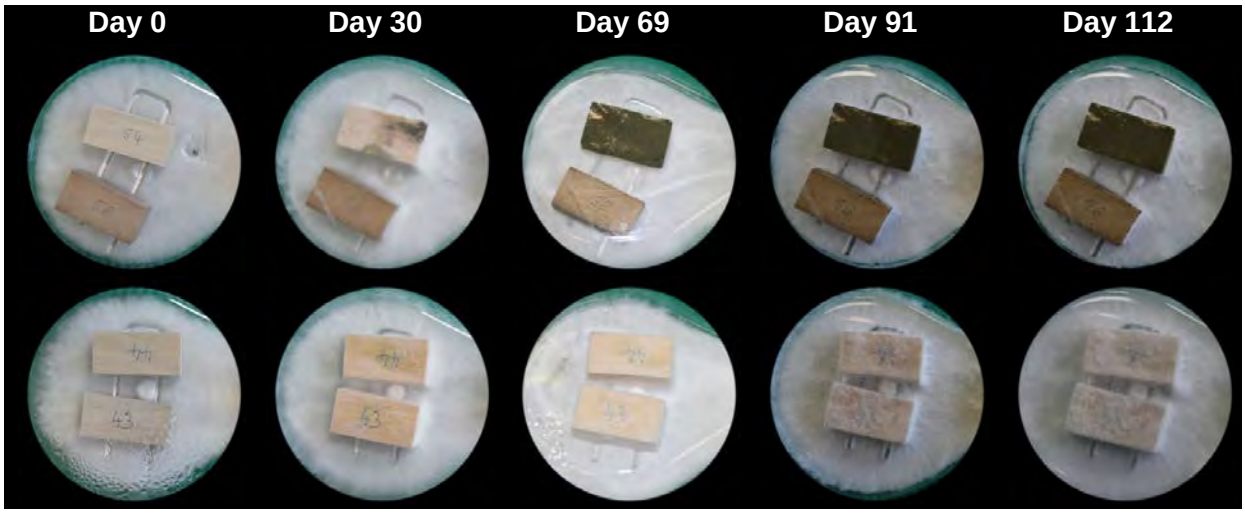

Fig. 2.
Photo series of reference (above) and virulence (below) samples' fungi resistance (Poria placenta).

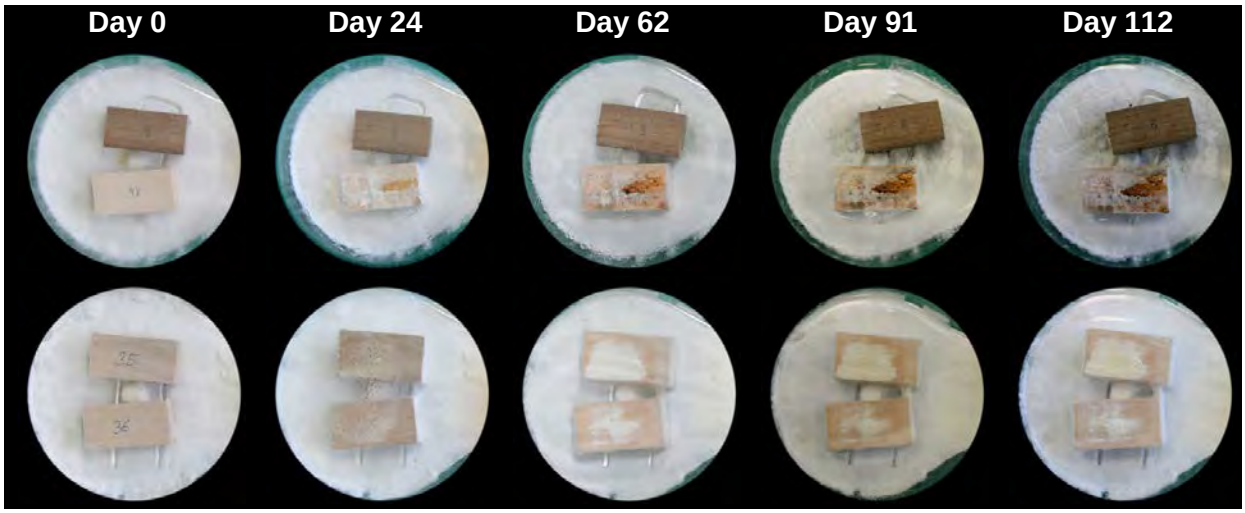

Fig. 3.
Photo series of reference (above) and virulence (below) samples' fungi resistance (Coriolus versicolor).

Long-term field test

The durability class was determined for each sample according to EN 252 (Table 2). After 6 and 12 months, there was no sign of decay on the acetylated samples and they dried shortly after being taken from the soil. Hornbeam was attacked by insects as there were holes present on each sample, and the surface was softened. The beech samples had similar rate of decay. The pine sapwood samples had insect's holes, softened surface, especially in earlywood and signs of white mould. According to the results so far, acetylation greatly improves the durability of hornbeam against mould, fungi, insects and moisture (Fig.4.).

Table 2
Durability classes of long-term field test samples in ground contact after 1 year according to EN 252. (H: hornbeam, AC: acetylated hornbeam, B: beech, PS: pine sapwood, AVG: average)

	1	2	3	4	5	6	7	8	9	10	11	12	AVG
H	2	4	4	4	2	2	2	2	2	2	2	2	**2,5**
AC	0	0	0	0	0	0	0	0	0	0	0	0	**0**
B	2	2	2	2	2	4	2	2	2	2	2	2	**2,2**
PS	3	3	3	3	3	3	3	3	3	3	3	3	**3,0**

Fig. 4.
Palcement of stakes in long-term field test (up) and stakes taken out for evaluation (bottom).

CONCLUSIONS

Acetylation prevented all three fungi species from attacking hornbeam which in its natural state is a non-durable wood species (Class 5 according to EN 350). After being exposed to fungi for 16 weeks, the treated samples' weight loss was below 1%, which makes it a very durable material (Class 1 according to EN 350). The presence of the acetylated material also had an impact on the reference samples, as these were less decayed by fungi than the virulence specimens.

So far in the long-term field tests, acetylated hornbeam shows great resistance against the fungi, mould, insects and moisture compared to hornbeam, beech and pine sapwood. These tests are continuously evaluated every 6 months.

Acetylation is an environmentally-friendly way to improve the durability of hornbeam, which could widen this species' usage in outdoor applications.

ACKNOWLEDGEMENT

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DRYING TIME AND QUALITY OF EDS-TREATED COMPARED TO UNTREATED BEECH WOOD (*Fagus japonica*)

Mihaela CAMPEAN
Prof.dr.eng. - Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: campean@unitbv.ro

Sachio ISHII
Manager - EDS Laboratory
Maebashi-shi, Gumma-ken 371-0101, Akagisan, Fujimi-machi 1863, Japan
E-mail: info@eds-lab.jp

Sergiu GEORGESCU
PhD Student - Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: sgiutz@unitbv.ro

Abstract

The paper presents the results of an experimental study performed with Japanese beech timber heat-treated by the EDS method (Japanese patent) and then dried in order to evaluate the effects of this treatment upon the drying rate and the drying uniformity. The obtained results demonstrate benefitting effects of the EDS treatment both upon the drying time and the drying quality. The drying rate of EDS-treated beech wood is by 29% higher in the case of wood without red heart and by 11% higher in the case of wood with red heart. As far as the drying uniformity is concerned, the minimum moisture content gradient across the 50mm thickness of the timber boards was recorded for the EDS-treated beech wood with red heart (ΔMC=1.66), by 21.7% lower than in the case of untreated beech wood with red heart.

Key words: *EDS treatment; beech wood; drying rate; drying uniformity; drying stresses.*

INTRODUCTION

EDS company was founded in 1984 as a timber manufacturer. Its main concern is directed towards the protection of the natural environment in all its richness, especially towards the protection of forests. This strategy was put into practice by developing several sollutions for the more rational use of wood, the valorization of inferior wooden resources and cellulosic plants which were not targeted for industrial uses until now, as well as a recycling-oriented use of wooden resources.

One of the main contributions of EDS company is an innovative technique (Ishii 1991) of heat-treating wood as a log in order to change, by means of high temperature (70-200°C) and smoke, its chemical composition so as to improve some of its properties.

The technique has proven its efficiency on low-value species from Asia (*e.g. Acacia mangium*, *Albizia falcataria*, palm wood, rubber tree wood, bamboo, coconut timber) (http://www.eds-lab.jp/english/jyumoku.html). The present research, in frame of a contract concluded between Transilvania University in Brasov and EDS Laboratory, pursues the effects of the EDS treatment upon Japanese beech wood (*Fagus japonica*), envisaging in case of positive results, a potential valorization of this technique on European beech (*Fagus sylvatica* L.) as well.

OBJECTIVE

The main objective of the study presented in this paper was to evaluate the drying time and drying quality of Japanese beech wood (*Fagus japonica*), with and without red heart, after being treated by the EDS method (Ishii 1991), comparatively to untreated wood originating from the same tree. The influence of the red heart presence was also pursued.

MATERIAL, METHOD, EQUIPMENT

The wooden material used within the experiments consisted of 50mm thick timber pieces originating from two logs of Japanese beech: one with red heart (average oven-dry density: 640kg/m^3), the other one without red heart (average oven-dry density: 626kg/m^3).

Half of the timber pieces from each log were first heat-treated by the EDS method in the EDS kiln (Fig. 1) at Maebashi-shi (Japan). The maximum temperature inside the kiln during the treating process was 150°C. The temperature inside wood was monitored continuously. It was raised up to 80°C and then maintained at this level for 72 hours, then cooled down to 50°C for further 48 hours.

Fig. 1.
EDS kiln (http://www.eds-lab.jp/english/puranto.html)

Afterwards, all pieces were conditioned for 2 months under constant parameters, at 20°C and 55% RH and then shipped to Romania.

As soon as they arrived at the University in Brasov, the moisture content of the timber pieces was measured by means of a Brookhuis capacitive moisture meter, in six positions, as shown in Fig. 2, to cover as best as possible the timber length, but also to take into consideration the differences that may appear between the centre and the exterior part of the timber board.

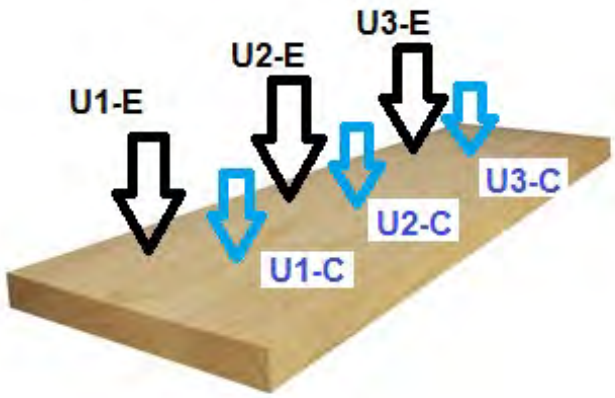


Fig. 2.
Assessing the moisture content of the timber boards, as average of six values, measured over timber length (1-2-3), in central part (C) and exterior part (E) of the board.

Then the boards were stacked in order to be dried in a conventional kiln with automatic kiln control. Eight boards (two of each assortment) were selected and introduced in rows 4 and 11, so as to have one piece of each assortment in comparable positions within the stack (Fig. 3). The moisture content of wood was monitored in these eight pieces, by means of resistive V2A sensors.

Fig. 3.
Positioning of wood moisture content monitoring sensors within the stack.

The applied drying schedule is presented in Table 1.

Table 1

Drying schedule

Phase	Wood moisture content, %	Air temperature, °C	Equilibrium moisture content, %
Warming-up	MC_{inital}	30°C	14%
Actual drying	$MC_{initial}$ ... MC_{final}	gradual raise up to 52°C	Gradual decrease down to 2.7%
Cooling	MC_{final}	-	-

Based on the difference between the initial moisture content (before drying) and the final moisture content (after drying), the drying rate (*w*) was calculated for each monitored board:

$$w = \frac{MCi - MCf}{D} \quad [\% / h] \quad (1)$$

where:
MC_i – moisture content of the board at the beginning of actual drying, in %;
MC_f – moisture content of the board at the end of actual drying, in %;
D – duration of actual drying.

Hereinafter, the timber pieces were subjected to the final quality control. Establishing the moisture gradient over the timber thickness (by means of layer-samples) and also the level of inner stresses (by means of fork-samples)(Fig. 4) were the main envisaged tasks.

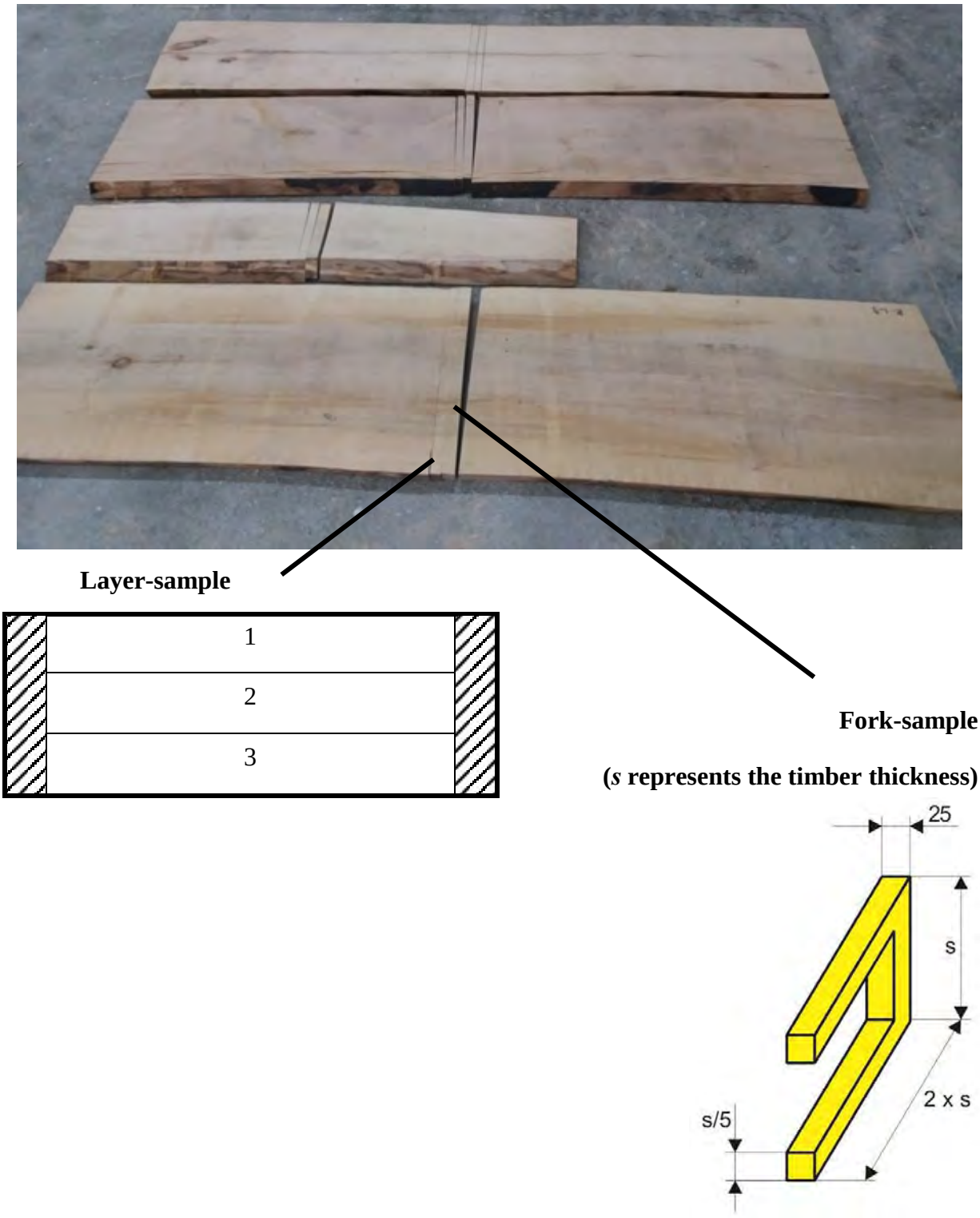


Fig. 4.

Layer-sample and fork-sample used within the quality control after the drying process for the determination of the moisture content uniformity over the timber thickness and of the internal stresses due to drying.

The layers (1-2-3) were weighed first right after drying (*m*), then again after oven-drying at 103°C (m_0). An electronic scale by KERN with a precision of 0.001g and an electric oven by BINDER were used to this purpose. The moisture content of each layer was calculated according to the relation:

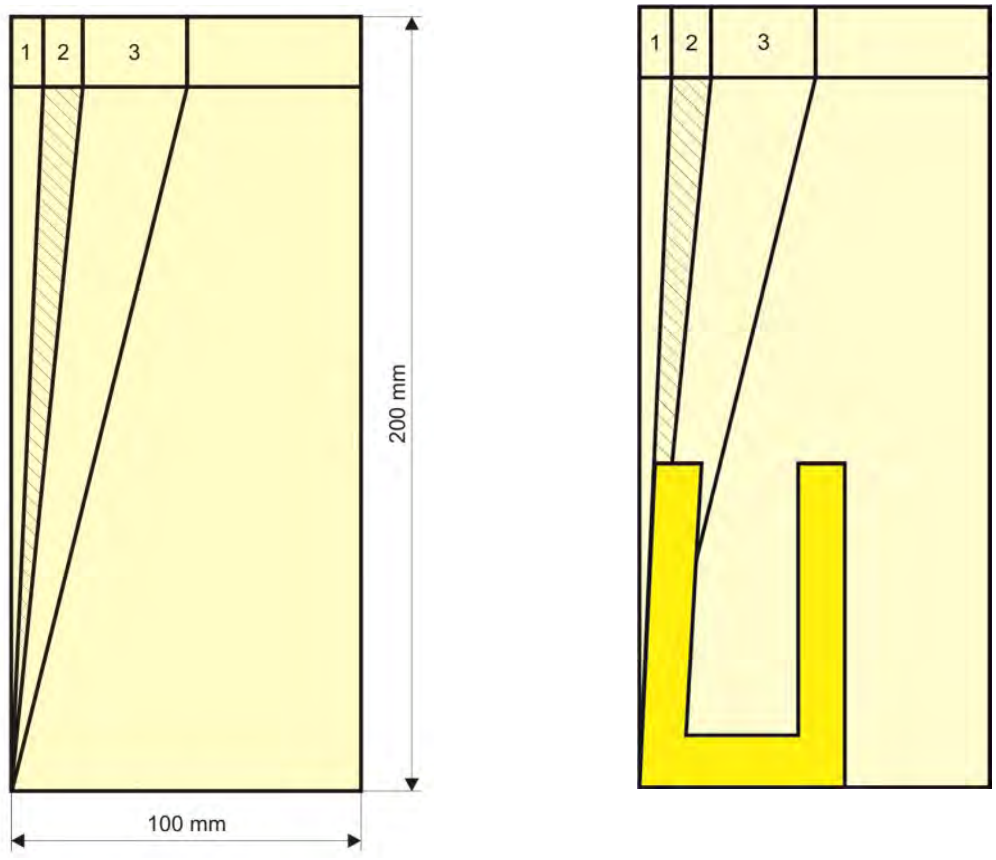
$$MC = \frac{m - m_0}{m_0} \cdot 100 \quad [\%] \quad (2)$$

Then the moisture content gradient (ΔMC) across the timber thickness was calculated as difference between the highest and the lowest value obtained from each sample:

$$\Delta MC = MC_{max} - MC_{min} \quad [\%] \quad (3)$$

ΔMC represents a valuable indicator of drying quality: values higher than 5%, indicate a high level of internal stresses, and thus, high risk of checking.

In order to determine the level of internal stresses, the fork samples cut according to the dimensions indicated in Fig. 4 were placed on an evaluation jig (Trübswetter 2006)(Fig. 5).



a. ***b.***

Fig. 5.
Determination by means of fork-samples of the internal stresses caused by drying:
a – evaluation jig; b – correct positioning of the fork-sample on the jig.

The average curving degree of the two teeth, placed at turn in the corner of the jig determine the gravity of the stress degree: 0...1-very low stress; 1...2-normal stress; 2...3-high stress; >3-very high stress.

RESULTS AND DISCUSSION

The results concerning the drying rate of EDS-treated and untreated Japanese beech with and without red heart are presented in Table 2.

Table 2

Drying rate values for Japanese beech timber

Wood type / MC sensor (row)	**Initial MC, %**	**Final MC, %**	**Drying rate per sample, %/h**	**Average drying rate per wood type, %/h**
Untreated wood, without red heart				
S2 (row 4)	9.4	7.5	0.026	**0.038**
S6 (row 11)	11.8	8.2	0.050	
EDS-treated wood, without red heart				
S4 (row 4)	10.5	8.2	0.032	**0.049**
S5 (row 11)	12.6	7.8	0.067	
Untreated wood, with red heart				
S3 (row 4)	9.3	7.3	0.028	**0.036**
S7(row 11)	10.7	7.5	0.044	
EDS-treated wood, with red heart				
S1 (row 4)	10.0	7.6	0.033	**0.040**
S8 (row 11)	10.8	7.5	0.046	

It can be noticed that, in all cases, the drying rate recorded for the samples in row 11 was higher (almost twice) than of those of the same wood type, but situated in row 4. The average values per wood type clearly show that EDS-treated wood dries faster than untreated wood. The drying rate is by 29% higher in the case of wood without red heart and by 11% higher in the case of wood with red heart.

Table 3 presents the results concerning the moisture gradient over the timber thickness.

Table 3

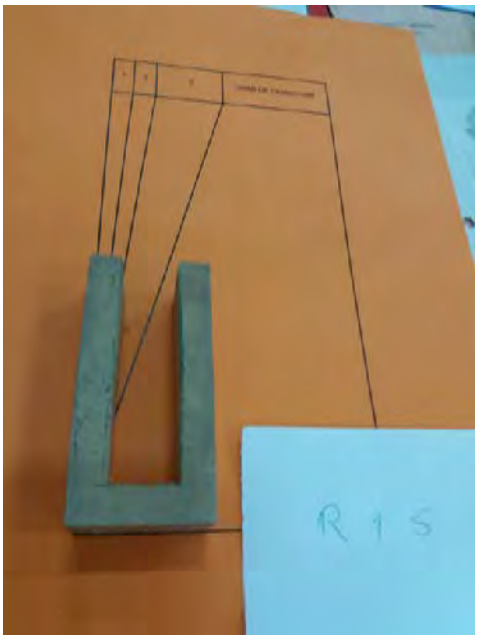
Moisture gradient across the timber thickness, after drying

Wood type	Mass of each layer right after cutting, before oven-drying, g	Oven-dry mass of each layer, g	Moisture content of each layer after the drying process, %	Moisture content gradient across timber thickness (ΔU), %
Untreated wood, without red heart	43.72	39.91	9.55	2.07
	41.55	37.47	10.89	
	38.50	35.38	8.82	
EDS-treated wood, without red heart	31.41	28.93	8.57	1.78
	28.78	26.18	9.93	
	26.26	24.28	8.15	
Untreated wood, with red heart	56.78	51.67	9.89	2.12
	63.19	56.64	11.56	
	49.94	45.63	9.45	
EDS-treated wood, with red heart	52.90	48.48	9.12	1.66
	53.73	48.55	10.67	
	45.84	42.05	9.01	

The results presented in Table 3 confirm the fact that EDS-treated wood dried more uniform than untreated wood: the moisture gradient across timber thickness is by 14% lower in the case of EDS-treated beech wood without red heart and by 21.7% in the case of EDS-treated beech wood with red heart. Considering that beech wood with red heart is known as more unhomogenous, as also demonstrated in this study by the highest value obtained among the four wood types (Table 3), the benefit of the EDS treatment becomes more significant: EDS treatment succeeds in „calming down" red heart beech wood and to ensure a uniform drying of this difficult wood type. This finding is also supported by the results obtained by means of the fork-samples (Fig. 6).

Untreated wood without red heart: 1.1 / 1.0 (average = 1.05)

EDS-treated wood without red heart: 0.8 / 0.5 (average = 0.65)



Untreated wood with red heart: 0.1 / 1.1 (average = 0.6)

EDS-treated wood with red heart: 0.4 / 0.5 (average = 0.45)

Fig. 6.
Experimental results regarding the internal stresses caused by the drying process, obtained with fork-samples.

CONCLUSIONS

According to the obtained results, the EDS treatment has benefitting effects both upon the drying time and the drying quality of Japanese beech wood. The effects are more visible in case of wood with red heart, which is normally more unhomogenous and more difficult to dry than beech wood without red heart. Under the effect of EDS treatment it dries faster than untreated wood without red heart and more uniform than all other considered wood types.

ACKNOWLEDGEMENT

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MOR AND MOE OF SOLID WOOD PANELS MADE FROM HEAT-TREATED BEECH WOOD

Murat HACIBEKTASOGLU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: murathacib@hotmail.com

Mihaela POROJAN
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: mporojan@unitbv.ro

Sergiu GEORGESCU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: sgiutz@unitbv.ro

Mihaela CAMPEAN
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: campean@unitbv.ro

Abstract

The paper presents the results of an experimental research performed with solid wood panels, made from heat-treated and untreated beech wood (Fagus sylvatica L.) strips. Specific samples were cut in longitudinal and transversal direction of the panel, in order to determine their mechanical properties in bending (MOR and MOE). The results show a reduction by 41% (in longitudinal direction) and by 103% (in transversal direction) of MOR for the panels made from heat-treated wood compared to the ones made from untreated wood. The MOE of the panels made from heat-treated strips did not show a noticeable modification in longitudinal direction, but it was by 15.7% lower in transversal direction, as compared to the one of panels made from untreated strips. As far as the influence of the cutting direction is concerned, the values of MOR and MOE in longitudinal direction are close to the ones given for solid wood. In transversal direction, the values of MOR and MOE are more than 10 times lower than in longitudinal direction.

Key words: *solid wood panels; beech; MOR; MOE.*

INTRODUCTION

It is a well-known fact that a heat treatment at temperatures above 180°C reduces the mechanical strengths of wood. The effects of different heat treatments (using various treating environments, different temperatures and times) upon the mechanical strengths of wood (*e.g.* tensile strength, compression strength, bending strength and modulus of elasticity, shock resistance, nails and screws withdrawal resistance, hardness etc.) were previously studied by many researchers. Most studies were performed with resinous wood, but a few studies refer to beech wood as well.

Ruschke (1973), cited by Esteves and Pereira (2009), made experiments with beech wood both in environments with and without oxygene, and concluded that the modulus of elasticity decreased significantly for mass losses from 8% to 10%.

Kamdem *et al.* (2002) used the French heat treating method (Rectified Wood). By heat-treating beech wood (*Fagus sylvatica* L.) at temperatures between 200°C and 260°C, the authors obtained a reduction of 20% for MOE and of 40% for MOR.

Wetzig *et al.* (2011) compared in-between and also relative to untreated wood, several mechanical properties of beech wood (*Fagus sylvatica* L.), heat-treated in a nitogen atmosphere and in a superheated steam atmosphere. The results revealed that both the tensile strength and the compression strength were reduced by the heat treatments, the reduction being more evident for the samples treated in a nitrogen atmosphere (e.*g.:* the tensile strength decreased from 150.09 N/mm^2 - for the untreated wood - to 53.5 N/mm^2 for wood treated in nitrogen and 84.39 N/mm^2 for wood treated

in superheated steam, respectively). Some elasticity moduli, like the E-module in tension and the G-module in dynamic shearing increased as compared to untreated wood.

A similar study was performed by Candelier *et al. (*2013), who evaluated the effect of the inert atmosphere, nitrogen versus vacuum, on the weakening of mechanical properties occurring during wood thermal treatment. The results showed that wood heat-treated under nitrogen presents lower MOR and MOE in bending and lower Brinell hardness comparatively to wood heat-treated under vacuum. Of the three mechanical properties investigated, MOR was the most sensitive property to the heat treatment conditions.

Todorovic *et al.* (2012) established correlations between the colour change (luminosity change ΔL and total colour change ΔE) and some physical and mechanical properties of beech sapwood and beech red heart (e.g.: a linear regression function with R^2=0.76 was found between ΔL and MOR for beech sapwood, heat-treated in a laboratory chamber at 170°C, 190°C and 210°C for 4h).

In her doctoral thesis, defended at the Transilvania University of Brasov, Olarescu (2015) studied the correlation between mass loss and dimensional stabilisation for several European species, heat-treated in a laboratory oven, at atmospheric pressure, at temperatures between 180°C and 200°C for 1h to 6h. The optimum heat-treating schedule for beech was found to be 200°C/3h, so as to obtain high dimensional stability (above 50%), without exceeding a mass loss of 5%.

A further step was taken by the first author of the present research, who studies within his doctoral research, the mechanical and technological properties of panels made from heat-treated beech wood strips.

OBJECTIVE

The main objective of the present research was to establish the mechanical properties in static bending (*MOR* and *MOE*) of solid wood panels made from heat-treated beech wood (*Fagus sylvatica* L.) strips compared to panels made from untreated beech wood.

MATERIAL, METHOD, EQUIPMENT

The wooden material used within the experiments consisted of 1600 × 800 × 17 mm solid wood panels (Fig. 1), made from heat-treated and untreted beech wood (*Fagus sylvatica* L.) strips, respectively.

a. ***b.***

Fig. 1.
Solid wood panels made from heat-treated (a) and untreated (b) beech wood strips.

The wood strips were heat-treated in superheated steam in an industrial-scale TekmaWood kiln, at JFF FURNIR Brasov, according to the schedule presented in Table 1. The average mass loss due to this heat treatment was 13.18% ± 1.36%.

Table 1

Heat-Treatment Schedule

Phase	Conditions (Temperature / Time)
Warming Up	100 °C / 3 h
Heating	100 °C…200 °C / 21 h
Actual Heat Treatment	**200 °C / 2.5 h**
Cooling	200 °C…30 °C / 13.5 h
Total Process Duration	**40 h**

The panels were manufactured by fingerjointing the strips in length and using a water-based PVAc adhesive DORUS MD 076/26 (D4) for the edge-to-edge gluing.

The sampling of the panels for the bending test (Fig. 2) was performed according to EN 326-1:1994, by cutting out ten longitudinal (Fig. 3, a) and ten transversal (Fig. 3, b) samples from each panel, sized at 390 x 50 x 17mm.

The testing was performed according to EN 310:1993 on an IMAL model IBX 600 equipment (Fig. 4). The machine records the maximum force when rupture occurs (F_{max}) and, based on this, the bending strength (*MOR*) can be calculated according to Equation (1):

$$MOR = \frac{3 \cdot F_{\max} \cdot l_1}{2 \cdot b \cdot t^2} [N/mm^2] \qquad (1)$$

where: F_{max} – force that produces rupture, in N;
l_1 – distance between the bearings, in mm;
b – sample width, in mm;
t – sample thickness, in mm.

and the modulus of elasticity (*MOE*) is calculated according to Equation (2):

$$MOE = \frac{l_1^2 \cdot (F_2 - F_1)}{4 \cdot b \cdot t^2 \cdot (a_2 - a_1)} [N/mm^2] \qquad (2)$$

where: $(F_2 - F_1)$ is the increase of the force along the straight part of the load-deformation curve;
$(a_2 - a_1)$ – deflection increase at half sample length, corresponding to $(F_2 - F_1)$.

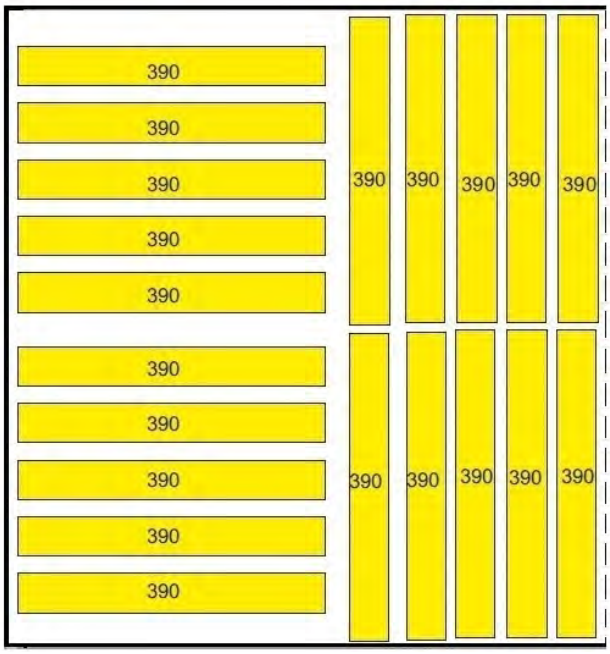

Fig. 2.
Sampling of solid wood panels for bending test.

a. ***b.***

Fig. 3.
Longitudinal (a) and transversal (b) panel sample for bending test.

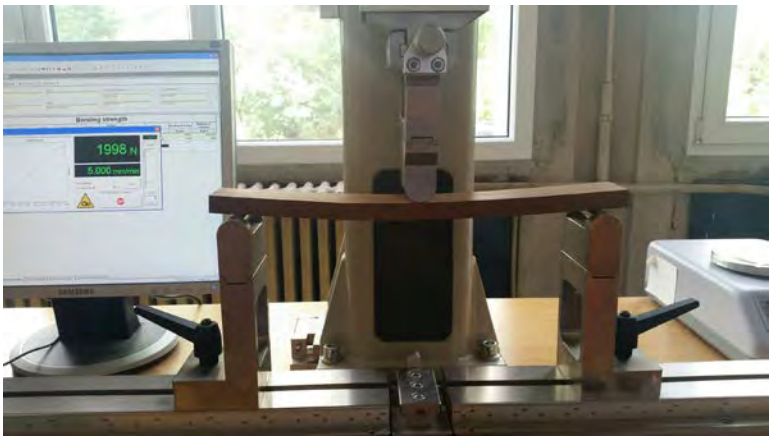

Fig. 4.
IMAL model IBX 600 equipment for determination of MOR and MOE.

RESULTS AND DISCUSSION

The results concerning the bending properties of the solid wood panels made from heat-treated and untreated beech wood strips are presented in Table 2.

Table 2

MOR and MOE of panels made from heat-treated and untreated beech wood strips (mean value ± standard deviation)

Beech wood	*MOR*, N/mm^2		*MOE*, N/mm^2	
	longitudinal	transversal	longitudinal	transversal
Untreated	83.26±25.56	5.71±1.13	12771±1322.78	908±107.81
Heat-treated	59.03±25.07	2.81±0.70	13000±1424.83	784±93.74

As shown in Table 2, the *MOR* and *MOE* values are much higher in case of the longitudinal samples. This result was expected, considering that these samples are very close to solid wood. Indeed, the values recorded for the *MOR* and *MOE* of the untreated longitudinal samples fit well into the ranges given by Holzatlas (2008) for solid beech wood: *MOR* = 74...123...210N/mm^2 and *MOE*=10000...16000...18000N/mm^2. For the heat-treated longitudinal samples, the bending strength is 41% lower. This result is in good accordance to the one obtained by Kamdem *et al.* (2002). The modulus of elasticity, however, seems not to be significantly influenced by the heat treatment.

In case of the transversal samples, *MOR* and *MOE* decrease severily (more than 10 times) both for the untreated, and for the heat-treated wood. This is due to the numerous gluing lines (originating from the edge-to-edge gluing of the wood strips in order to create the panel width) along each sample. The weakening is more evident for the heat-treated samples, which is due to their hygroscopicity which acts repellant not only upon moisture, but also upon the water-based adhesive.

The rupture (Fig. 5) occurred in wood at all longitudinal samples (Fig. 4, a), both in case of the heat-treated and the untreated wood. With the transversal samples (Fig. 4, b), the rupture occurred in wood (along the rays) in case of the most samples from the heat-treated panel, which demonstrates that the wood weakening effect due to the heat treatment is stronger than the water repellance effect. The transversal untreated samples were the only ones where the rupture occurred very close to the gluing line.

a. ***b.***

Fig. 5.
Rupture caused by static bending in panel sample: a-longitudinal sample; b-transversal sample.

CONCLUSIONS

The conclusions of the present research can be formulated as follows:

1. The bending strength of solid beech wood panels made from heat-treated strips is by 41% lower in longitudinal direction and by 103% lower in transversal direction than that of panels made from untreated strips.
2. The modulus of elasticity in bending of panels made from heat-treated strips did not show a noticeable modification in longitudinal direction, but it was by 15.7% lower in transversal direction, as compared to the one of panels made from untreated strips.
3. By analysing comparatively the rupture mode of the samples, it was noticed that in most samples the rupture occurred in wood. The transversal untreated samples were the only ones where the rupture occurred very close to the gluing line.
4. As a general conclusion after this test, referring to the influence of using heat-treated wood strips in solid beech wood panels, one can conclude that the strength reduction in bending is considerably high, and so, a less invasive heat treatment (lower mass loss) should be applied if the panel use implies bending loads (***e.g.*** for garden tables or benches).

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COLOUR MODIFICATION OF WOOD IN SIMULATED SUNLIGHT EXPOSURE

Dan ROSU
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: drosu@icmpp.ro

Liliana ROSU
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: lrosu@icmpp.ro

Teodora RUSU
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: teia@icmpp.ro

Carmen-Alice TEACĂ
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: cateaca@icmpp.ro

Cristian-Dragos VARGANICI
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: varganici.cristian@icmpp.ro

Abstract

The light stability of wood panel treated with maleic anhydride at different concentration values was investigated. Analysis of the colour changes in wood surfaces during artificial light irradiation was carried out by measuring CIELAB parameters. Fourier transform infrared spectroscopy was used to study chemical changes caused by photo-degradation. The colour difference of maleated wood after irradiation were slightly less than those of the control sample, indicating that esterification inhibited to a less extent the photo-yellowing of wood. Maleic anhydride treatments reduced to a less extent the amount of the chromophore groups generated on the surface of irradiated softwood, with a lower effect on the photo-discoloration of wood.

Key words: *wood; photodegradation; colour modification.*

INTRODUCTION

Wood is a renewable and environmentally friendly bioresource and an increased use of material from renewable sources is highly desired for a sustainable development. It is an important material available from sustainable source that may be composted, incinerated and recycled at the end of product life-cycle. Although the wood is a versatile material used in many applications such as building, construction and furniture, there are some drawbacks in the practical use, such as: the low durability of many species, the dimensional instability with change in moisture content, the low resistance against fungi and to the attack of insects etc. The photo-yellowing and inappropriate

mechanical strength properties are observed when the wood is exposed outdoors (Hon 1991). Contrary to these disadvantages people want to maintain the original characteristics of natural wood. In many applications, we are interested in achieving a stable colour, rather than necessarily maintaining the initial colour for example, for furniture, decorative panels and flooring. The photo-degradation process of wood involves very complex physical and chemical reactions. There have been many studies attempted to clarify the mechanism of wood weathering (Hon 1983; Hon et al. 1984; Pandey 2005). It has been shown that the degradation process is triggered by the formation of free radicals during UV irradiation (Muller et al. 2003). Despite many studies carried out several decades, the precise mechanism involved in the photo-degradation of wood is not well defined. Modification of wood is a commercially available practice that has a long history demonstrated by the plethora of uses within our society. Prevention of photodegradation by wood modification offers protection of wood (either with or without a coating) and seems therefore an attractive option for exterior wood use. Two primary categories for wood modification are esterification or etherification (Matsuda 1996; Chang et al. 2002). Esterification is the process of converting the hydroxyl group into an ester group; with wood this is usually done through the nucleophilic addition of an organic acid anhydride or acid chloride. Esterification of wood has primarily focused on the use of anhydrides, with the exception of fatty acids and acid chlorides (Thiebaud et al. 1995) to improve dimensional stability, preservation, and wood thermoplastic-matrix compatibility (adhesion and dispersion). A variety of anhydrides with different catalysts and solvents have been used for these applications including acetic anhydride, maleic anhydride, phthalic anhydride, succinic anhydride, and other functionalized anhydrides. Durability of wood is important and can be addressed by esterification. To enhance the photostability of wood, anhydrides (acetic, succinic, maleic, phthalic) were reacted with wood to increase the number of chromophores (Thiebaud et al. 1995). An increase in succinic anhydride concentration may have a favorable effect on the succinoylation, as it was shown in the case of ultrasound irradiated sugarcane bagasse (Liu et al. 2008).

OBJECTIVE

The objective of the present study was to investigate the photo-discoloration and photo-degradation of the fir wood (*Abies alba*L.), which is widely used in Romania. For comparison purpose, the effect of chemical modification by treatment with succinic anhydride at different concentration values was investigated to present the differences in degradation performance. The degradation was monitored by measuring colour and chemical changes due to UV light irradiation.

MATERIAL, METHOD, EQUIPMENT

Prior to the chemical modification, the wood samples were extracted 8 hours with xylene. The extraction was performed in a Soxhlet apparatus, for the decrease the material extractives influence on the chemical modification of the softwood. The wood extracted samples were dried 24 hours in an oven at 70°C up to obtain a constant weight. For esterification, the dried wood samples were dipped in succinic anhydride (SA) dissolved previously in xylene and heated at 90°C under continuous stirring for 7 minutes. The concentration levels of SA in the solution were designed to be 0 g/L (etalon sample), 10 g/L (sample 1), 30 g/L (sample 2) and 60 g/L (sample 3). After esterification the specimens were removed from the solutions and cooled to room temperature. A new extraction with xylene for 8 h was performed in order to remove the non-reacted SA from the samples and finally the specimens were oven-dried for 24 hours at 70°C to reach a constant weight.

Samples thickness was monitored with a PosiTector 6000 device (De Felsko USA). Colour analyses were conducted with a Pocket Spec apparatus purchased from Colour QA SUA having a sensor head of 6 mm in diameter. The device was calibrated with a super white sulphate barium pellet. Measurements were conducted under reflectance mode using D65 illuminant at 10^0 standard observer. Results were extracted in the CIELAB system. The colour parameters of this system are the following: the L* axis is the lightness (ranging from 0 (black) to 100 (white)), whilst a* and b* axes represent the chromaticity coordinates (a positive a* value corresponding to red and a negative a* value to green, whilst +b* and -b* denote yellow and blue, respectively). The FT-IR spectra were recorded using a Bruker Vertex 70 device equipped with a MIRacle accessory designed for single or multi-reflection attenuated total reflectance (ATR).

UV radiation was conducted in air by means of a rotating hexagonal prism device, used as sample carrier, having a light source on the device's central axes. A medium pressure Hg vapour lamp, model OSRAM HQE-40 Hg (Germany), of 100 W power, with a polychrome emission spectrum between 240 and 570 nm, a maximum at 365 nm and a filter for wavelengths with $\lambda > 280$ nm, was the artificial light source, having an average irradiance value of 95 W m^{-2} and an average hourly exposure

dose of 350 kJ m^{-2}. Irradiance and exposure dose values were determined with a A PMA 2100 radiometer having a UVA detector type PMA 2110, from Solar Light Co. (USA).

RESULTS AND DISCUSSION

In contrast to other anhydrides, SA is not able to swell the softwood structure. Therefore, a solvent should be added to the reaction system to increase the accessibility of the reactive hydroxyl groups of lignin, hemicelluloses, and cellulose in softwood to the reactions. Furthermore, to obtain esterified softwood bearing carboxyl groups, the SA should be used in the liquid state by dissolving in xylene solvent (SA has a melting point of 119–120°C). The polar solvent like pyridine is toxic, has an unpleasant odour and is not suitable to use in a large scale reaction even though it is an effective catalyst in such acylations (Hill et al. 2000). Non-polar solvents like xylene, hexane leads to poor grafting efficiency, whereas polar solvent like N, N-dimethylformamide (DMF) gives satisfactory results. However, DMF is a expensive solvent for esterification reactions (Sun et al. 2004).

Identification of signals in FTIR spectrum

The FT-IR spectrum of *Abies alba* L. wood sample is shown in Fig. 1. The large characteristic absorption from 3373 cm^{-1} corresponds to O-H stretching vibration. The C-H stretching vibration in methyl and ethylene groups appeared at 2890 cm^{-1} and 2919 cm^{-1}. The large peak at 1730 cm^{-1} characterizes the carbonyl stretching vibration in non-conjugated ketenes and in free aldehyde present in lignin and hemicelluloses. The C=O stretch of conjugated or aromatic ketones absorbs below 1700 cm^{-1} and can be seen in Fig.1 as a large shoulder with the peak at 1653 cm^{-1}. The shoulder from 1604 cm^{-1} incorporates contributions from C=C unsaturated linkages, including aromatic rings present in lignin. The peak from 1509 cm^{-1} was assigned to the C=C stretching vibration in aromatic structure of lignin while the band from 1465 cm^{-1} characterize the asymmetric bending vibration of CH_3 group from lignin. The CH_2 bending vibrations related to the structure of cellulose appeared in the FT-IR spectrum of wood sample at 1421 cm^{-1}. The shoulder from 1371 cm^{-1} characterizes the O-H bending vibrations in phenols (lignin) and the signal from 1261 cm^{-1} is specific to the OH group from hydroxypropyl moiety (guaiacyl unit) in lignin. This observation was also suggested in other studies which shown that absorption band at around 1270 cm^{-1} indicates the presence of guaiacyl unit in the softwood lignin (Bodirlau et al. 2013). The shoulder from 1153 cm^{-1} is specific to the asymmetric bridge stretching vibration of C-O-C group in the structure of cellulose and the band from 1024 cm^{-1} was assigned to the symmetrical C-O stretch. Also, the signal from 897 cm^{-1} characterizes the glucomannan.

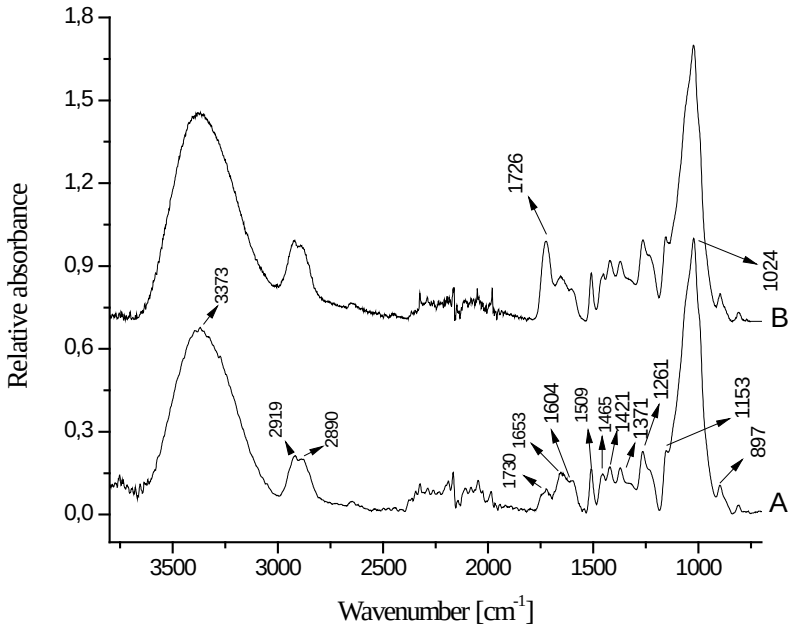

Fig. 1.
FT-IR spectra: (A) etalon sample (Abies alba wood); (B) sample 3 (Abies alba wood sample esterified with succinic anhydride).

A problem in ester analyses with FT-IR is the overlapping in wavelengths at around 1730 cm^{-1} where both ester carbonyls (C=O) and carboxyl carbonyls (C=O) absorb. There are studies in which the authors argue that absorption bands don't overlap, that ester carbonyl stretching is between1729 and 1748 cm^{-1} and that peaks at 1710–1725 cm^{-1} arise from the carboxyl carbonyl stretching groups (Matuana et al. 2001; Bodirlau et al. 2013).

The photochemical decomposition of lignin, which is the most susceptible to photodegradation, in the etalon sample and in the esterified softwood sample (sample 3) can be seen in Fig. 2. At high irradiation times, the absorbance of lignin decreases less in the esterified sample than in the etalon one. This observation explains the less colour changes with the irradiation time of the esterified softwood samples by a smaller amount of degraded lignin.

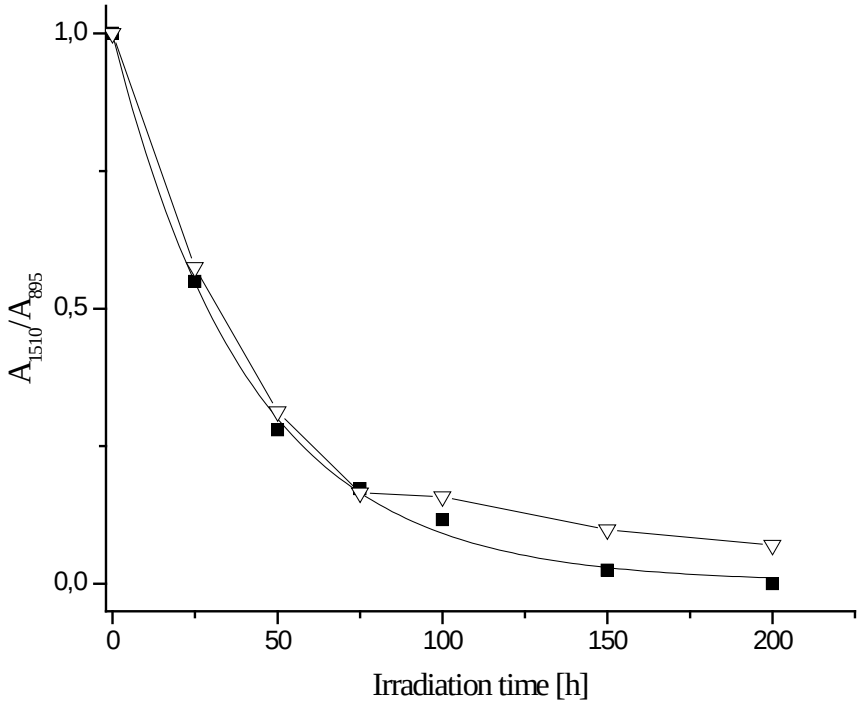

Fig. 2.
Variation of lignin content as a function of irradiation time measured by the ratio between the absorbance from 1509 cm^{-1} (A1509) and 897cm^{-1} (A897): (■) etalon; (□) sample 3.

Colour changes

In general, the total colour differences increased with irradiation time (irradiation dose). These values increased from 0 to +20.5 for non-modified wood (etalon sample) after 200 hours exposure time, from 0 to +19 for modified wood (sample 1 and sample 2) and from 0 to +12.5 for sample 3. Colour modifications of non-modified wood were explained by the increased values of chromatic parameters Δa^*, Δb^* and ΔL^*. The colour changes decreased with increasing of The SA concentration. ΔL^* values were negative for non-modified wood due to surface darkening during UV irradiation. This was due to lignin and non-cellulosic polysaccharides photo-degradation (Hon et al. 1984), hence irradiated samples were darker than the non-irradiated ones.

The increase of Δa^* and Δb^* values is due to quinoide-like structures resulted during lignin depolymerization and oxidation through free phenoxyl radicals (Hon 1991). This increase is also related to blue chromophores, formed during the first 50 hours irradiation time in all samples, with decreasing concentrations with of succinic ester content increase. The destruction of the blue chromophores occurred after 200 hours irradiation time, when increasing concentrations of yellow ones were generated. Δb^* values exhibited a decreasing trend for all samples. The esterified samples showed lower yellow chromophores concentrations.

CONCLUSIONS

Abies alba L. softwood surfaces were chemically modified by SA esterification and exposed to UV irradiation at a higher than 300 nm wavelength.

Structural modifications during UV irradiation were undertaken by FT-IR and discussed in correlation with colour changes.

The chemically modified softwood surfaces proved to be more stable to UV irradiation than the non–modified surface and the stability increased with increasing SA concentration.

ACKNOWLEDGEMENT

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STUDY OF UV IRRADIATION EFFECT ON WOOD SURFACE CHEMISTRY

Liliana ROSU
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: lrosu@icmpp.ro

Carmen-Alice TEACĂ
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: cateaca@icmpp.ro

Dan ROSU
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: drosu@icmpp.ro

Cristian-Dragos VARGANICI
Centre of Advanced Research in Bionanoconjugates and Biopolymers
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: varganici.cristian@icmpp.ro

Elena MARLICĂ
"Petru Poni" Institute of Macromolecular Chemistry
Gr. Ghica-Voda Alley 41A, 700487, Iasi, Romania
Tel: 0040 232217454, Fax: 0040 211299,
E-mail: emarlica@icmpp.ro

Abstract

Wood is a natural complex biocomposite material mostly comprised of lignin, cellulose and hemicelluloses. Due to its exposure in the natural environment, wood is susceptible to photooxidation from environmental factors. The consequences of wood exposure to the natural environment factors reside primarily on wood surface colour changes. An efficient path in overcoming this issue consists of coating the wood surface by protective layers through its chemical modification. The paper reports photostability evaluation of softwood surface subdued to chemical modification through successive treatments with succinic anhydride and epoxidized soybean oil, at a UV radiation wavelength above 280 nm. The photooxidation phenomena were monitored through colour changes and structural modification with irradiation dose and time. The chromatic coordinates values of modified wood surfaces showed a slow increase during UV irradiation as compared to those corresponding to unmodified wood. The epoxidized soybean oil coating layer proved to generate a highly efficient protective action of the chemically modified wood surface against photooxidation phenomena by generating a screening effect.

Key words: *wood; soybean oil; colour modification; photodegradation.*

INTRODUCTION

The existence of specific chromophores (aromatics, conjugated double bonds, phenols, etc.) within the wood structure increases its light absorbtion capacity. When exposed in the natural environment, wood mainly undergoes photooxidation processes through surface colour and gloss modifications and weakening of mechanical properties (Hon 2001).

The surface of wood deteriorates relatively fast when wood is exposed to the environment without any protection (Hon 2001; Hon 1984). It has been well established that sunlight (especially UV and visible light) and water are the most common elements damaging the wood surface during outdoor exposure [Hon 2001). Ligning proved to be the most affected by photodegradation through the yielding of free phenoxyl radicals, which further yield carbonyl and carboxyl groups through reactions with oxygen (Pandey 2005).

Degradation of cell wall polymers causes separation and strength loss of wood cells (Hon 2001). This can lead to the formation of micro-checks which can turn into surface cracks due to repeated swelling and shrinking of the wood (Hon 1984; Pandey 2005; George et al. 2005; Muller et al. 2003). These dimensional changes are a result of moisture uptake and drying. In addition, rain water leaches the photo-degraded wood fragments (mainly from lignin) bringing about greater surface roughness. After leaching of UV degradation products, underlying cell layers are exposed and further eroded. Two effective measures – surface coating and bulk treatment of wood – can, individually or conjointly, protect exterior wood from deterioration. Coatings present a physical barrier which protects the wood substrate from the adverse effect of environmental factors, such as solar irradiation, moisture, as well as staining and decay fungi. Clear coatings with high transparency require, however, additional UV absorbers and a radical scavenger, such as hindered amine light stabilizers to protect the coating itself and the wood surface underneath (Ahola 1991). Accordingly, it is assumed that certain pre-treatments of the wood substrate can improve the performance of exterior wood coatings through a dimensional stabilization, a reduction in capillarity and a greater fungal resistance.

Chemical modification reportedly renders wood dimensionally stable by depositing a chemical in the wood cell wall (bulking effect) and/or by cross-linking the cell wall polymers (Hill et al. 2000; Chang et al. 2006). Acetylation of wood was shown to improve the weathering and coating performance (Evans et al. 2000). Epoxidized vegetable oils and their derivatives have been used for many commercial applications, ***e.g.***, as plasticisers and stabilisers in chlorine containing resins, as additives in lubricants, as components in thermosetting plastics, in cosmetics and pharmaceutical formulations, in urethane foams and as wood impregnants (Wu et al. 2000). Epoxy fatty acid compounds are obtained at industrial scale mainly by the peracid process (Rangarajan et al. 1995; Sinadinovic et al. 2001; Hill 2000). Soybean oil is a triglyceride that typically contains 14% stearic, 23% oleic, 55% linoleic, and 8% linolenic acid. Chemical modification of commercially available soybean oil, such as epoxidation, can enhance its properties (reactivity) for certain industrial applications. The epoxidized soybean oil (ESO), an epoxidized glycerol fatty ester, is extensively used in the plastic industry as a plasticizer to increase flexibility in poly (vinyl chloride) (PVC) products and as a stabilizer to minimize their decomposition.

UV wood surface irradiation with wavelengths in the range 280–400 nm may generate significant colour and structural modifications, i.e. lignin content decrease, cellulose content increase with high deterioration of mechanical properties (Deka et al. 2008). A great deal of research is therefore conducted for developing wood protective systems against photo-decomposition during outdoor exposure. This may be accomplished either by surface chemical modification or by dyeing or painting (Rosu et al. 2010).

OBJECTIVE

The purpose of the paper resides in evaluating the photostability of chemically modified softwood surface by successive treatment with succinic anhydride (SA) and epoxidized soybean oil during exposure to UV radiation higher than 280 nm.

MATERIAL, METHOD, EQUIPMENT

Wood specimens as discs with the diameter of 6.5 mm and the thickness 0.4 mm were prepared from dried logs of *Abies alba* L. (5 years old) dried at ambient temperature for 1 year and previously debarked. Wood samples were polished with sandpaper (400 P) prior to use, after cutting. Wood specimens comprise both earlywood and latewood. We have used the axial surface meaning the cross-section for our investigations.

Triethylamine (TEA) and xylene were purchased from Sigma-Aldrich. ESO was obtained by a method described in the literature (Mustata et al. 2011). The epoxidized soybean oil (ESO) (0.273/100 g^{-1} epoxy equiv.). The wood discs were extracted with xylene (8 hours), oven vaccum dried at 70^0C (24 hours) and pretreated in xylene with SA solutions of different concentrations (60, 80, 120% w/w). 10 samples were held as both reference and each concentration value (M60, M80, M120).

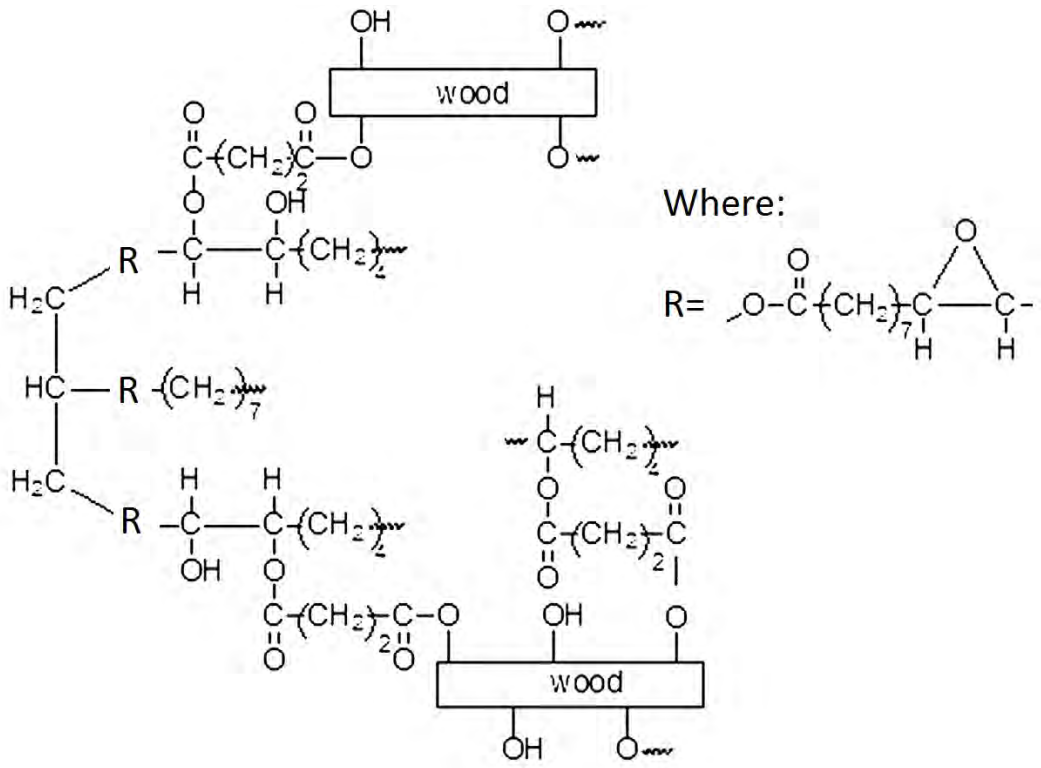

Fig. 1.
Wood treated with epoxidized soybean oil and succinic anhydride.

For samples thickness measurements a PosiTector 6000 device (De Felsko USA) was used. Colour analyses were conducted with a Pocket Spec apparatus purchased from Colour QA SUA having a sensor head of 6 mm in diameter. The device was calibrated with a super white sulphate barium pellet. Measurements were con-ducted under reflectance mode using D65 illuminant at 10^0 standard observer. Results were extracted in the CIELAB system. The colour parameters of this system are the following: the L* axis is the lightness (ranging from 0 (black) to 100 (white)), whilst a* and b* axes represent the chromaticity coordinates (a positive a* value corresponding to red and a negative a* value to green, whilst +b* and -b* denote yellow and blue, respectively). The FTIR spectra were recorded using a Bruker Vertex 70 device equipped with a MIRacle accessory designed for single or multi-reflection attenuated total reflectance (ATR).

The modified wood samples were exposed to UV radiation in air using a rotating hexagonal prism shaped device, as sample carrier, with the light source positioned on the central axis of the device. An OSRAM HQE-40Hg middle pressure lamp type (Germany) with 100W power, having a polychrome emission spectrum in the field 240 - 570nm, was used as artificial light source. The more energetic radiations with wavelengths below or equal 280nm, that are missing in the natural light spectrum, were eliminated with a quartz/borosilicate filter (30mm thickness) with maximum transparency at 365nm. Distance between samples and the lamp inside irradiation device was 60mm.

The average irradiance value at the sample surface was 95W m^{-2} and the average hourly exposure dose was 350kJ m^{-2}. A PMA 2100 radiometer provided with an UVA detector, type PMA 2110, manufactured by Solar Light Co. USA, was used for measurements of irradiance values and the exposure dose (time integral of irradiance).

RESULTS AND DISCUSSION

Colour changes

Colour modifications increased with irradiation time and dose, with the most significant ones occurring in the first 20 hours, the $\Delta E_{a,b}$ value reaching higher than 11 units for the unprotected woods and 5 units for the wood succinic monoester specimens, 3 units for the ESO treated wood surfaces and close to 0.2 units for ESO modified wood succinic monoesters. The human eye distinguishes colour modifications higher than 2 units. An efficient wood surface protective effect was observed after applying the SA and ESO, as opposed to unprotected wood surfaces, up to a UV irradiation dose of 700kJ m^{-2}. Also, this surface chemical modification led to a significant reduction in the colour modifications at higher irradiation dose values.

Identification of signals in FTIR spectrum

The reaction occurring between wood succinic monoester and ESO was monitored by ATR-FTIR spectroscopy.

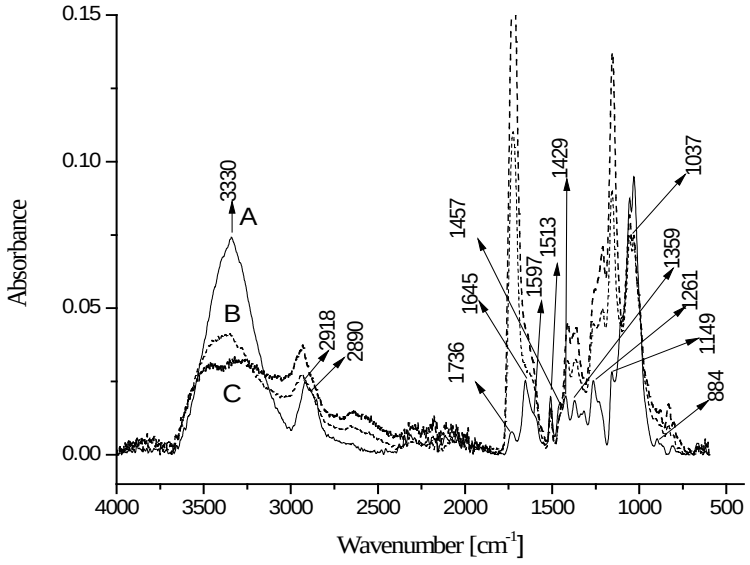

Fig. 2.
The FTIR spectra of non-modified wood sample (A), of the wood sample modified with succinic anhydride (B) and of the wood sample modified with succinic anhydride and coated with epoxidized soybean oil (C).

The band from 3330cm^{-1} characterizes the –OH stretching vibration from wood (hydrogen bonding). The vibrations from 2890 and 2918cm^{-1} characterize the C-H stretching in methyl and methylene groups. The signal from 1736cm^{-1} corresponds to carbonyl stretching vibration in non-conjugated ketones and in the free aldehyde present in lignin and hemicelluloses. The peak from 1645 cm^{-1} was assigned to the C=O stretching vibration of conjugated and aromatic ketones and the peak from 1597cm^{-1} corresponds to the C=C unsaturated linkages of aromatic rings present in lignin.

Also, the vibration from 1513cm^{-1} characterizes the C=C stretching vibration in the aromatic structure of lignin. The vibration from 1429cm^{-1} is specific to CH_2 bending related to the structure of cellulose and/or aromatic skeletal vibrations. The peak from 1359cm^{-1} characterizes the deformation vibration of C-H groups and O-H bending vibrations in phenols from lignin. The signal from 1261cm^{-1} was attributed to C-O vibrations of guaiacyl unit in lignin and the signal from 1149cm^{-1} was assigned to the asymmetric stretching vibration of C-O-C group in the structure of cellulose. Also, the vibration from 884cm^{-1} is specific to glucose ring stretching, C_1-H deformation and C-H stretching out of plane of aromatic ring.

The spectral changes confirmed the wood esterification with succinic anhydride. In the spectrum of the modified wood sample with succinic anhydride (spectrum B) the absorbance intensity from 3330cm^{-1}, specific to O-H stretching vibration from carboxylic moiety, decreases and enlarges to the lower wavelength. The band from 1736cm^{-1} significantly increases due to the new carbonyl group appeared after esterification. In addition, the intensity of absorption bands at 1429cm^{-1} assigned to the CH_2 was also enhanced after wood modification.

In the FTIR spectrum C, the signals from 2918, 2890 and 1736cm^{-1} increase due to the reaction between the succinic monoester with ESO.

CONCLUSIONS

SA and ESO modified *Abies alba L.* surface photostability was monitored during UV exposure with λ>280 nm by colour and structural modifications. Colour modifications increased with irradiation dose and time. SA and ESO protected the wood surfaces up to a irradiation dose of 700 kJ m^{-2}. The chemical modifications significantly reduced colour modifications.

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TESTING SOFA AS A SALES TOOL FOR THE SETUP OF INDIVIDUAL SIZE PARAMETERS OF SEATIGN FURNITURE

Ing. arch. Hynek MAŇÁK, PhD.
Mendel University in Brno, Department of Furniture, Design and Habitat
Zemědělská 3, 613 00 Brno, Czech republic
Tel: 00420 545 134 173, Fax: 00420 545 134 169, E-mail: hynek.manak@mendelu.cz

Abstract

The current trend in area of domestic seating furniture is the opportunity to choose individual size parameters of seating furniture: optional seating height and depth, optional height and width of armrests and optional seat firmness are available. For practical testing of specific optional parameters, it is necessary to try sitting on various seating furniture samples. A test sofa as a sales tool enables real testing of optional parameters and allows easy, comfortable and dignified selection of the optimal combination of seating furniture size parameters. The article describes specific optional seating furniture parameters of and presents proposals for technical solutions.

Key words: *seating furniture; ergonomy; individual size parameters; sales toll.*

INTRODUCTION

Seating furniture manufacturers offer the option of selecting individual parameters of the seating furniture. Commercially this is presented as custom furniture production (Promotion slogans: Polstermöbel im Wunschmass /manufacturer Dietsch, Mein Mass, Mein Sofa /manufacturer Koinor).

SEATING FURNITURE OFFER

Renowned German seating manufacturer Rolf Benz offers the selection of size parameters for most models: seat width, seat height, seat depth, height and width of armrests. The quality of the seat can also be influenced by the selection of seat hardness. The selection of leg type will not affect the seat ergonomy, but it affects the aesthetic appearance of the seating furniture.

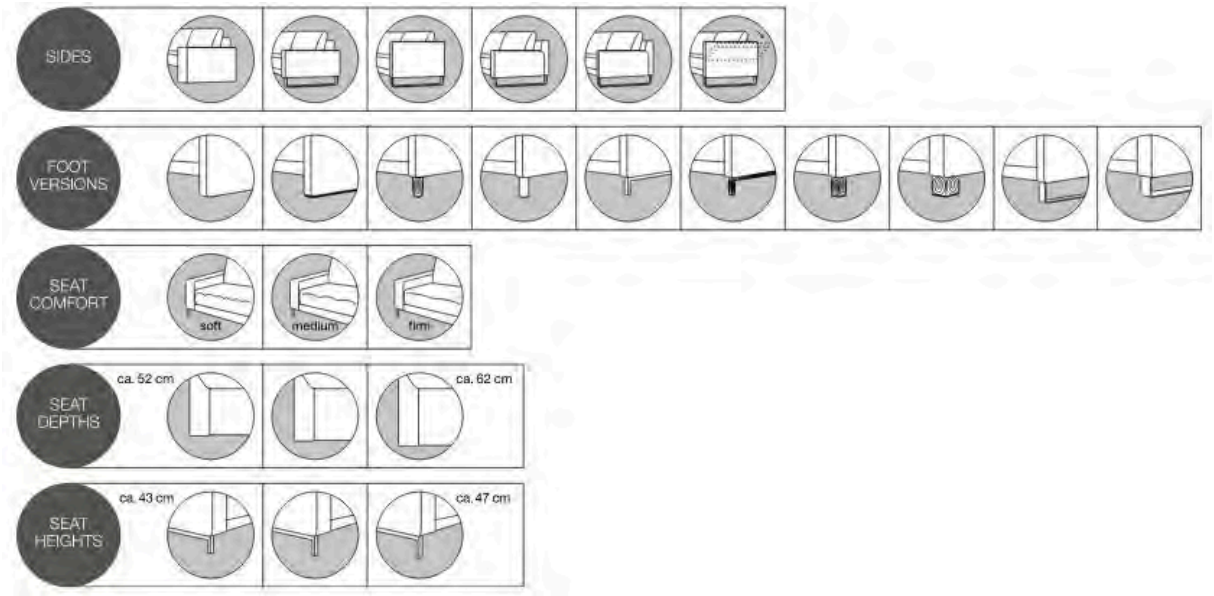

Fig. 1.
Optional size parameters of seating furniture Rolf Benz EGO [1].

Model Rolf Benz model 201 was for example offered with the seat in three heights, 43, 45, and 47cm. The armrests were available in 3 heights of relative height difference above the seat: low 8cm, medium 14cm and high 23cm. Some models of seating furniture even offer 4 armrest height variations with 2 selectable seat heights. The armrest of model 201 was available in 3 width variations, 10, 18 and 27cm. The mutual combination of 3 seat heights and 3 armrest heights along with 3 armrest width variations creates 27 armrest size variations.

The seat is available in up to 5 width variations within the overall width of the sofa, and in 2 depth variations, 55 and 62cm. Another optional parameter is the hardness of the seat - medium and hard, or selectable seat cushions. These size combinations are multiplied by the number of options of upholstery fabrics and leathers. The ordering system must clearly and unambiguously define these size combinations for production.

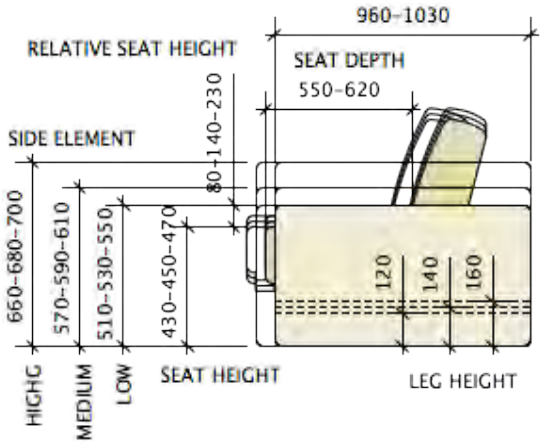

Fig. 2.
Optional size parameters of seating furniture Rolf Benz model 201.

With the mutual combination of selectable parameters, individual size parameters of the seating furniture can be selected for specific customers. It is important for customers to be able to make sense of this wide range of size parameters, in which trained sales staff may greatly assist them. For the right selection of seating furniture size parameters, it is best to try sitting on the sofa of the desired size.

MECHANIC TESTING ARMCHAIR

In the past, Rolf Benz used a testing armchair for these purposes, allowing mechanical adjustment and practical testing of the height and depth of the seat. A testing armchair also allows testing of the armrest height and width. The cushion hardness can be simulated by replacing the seat and backrest. The testing armchair was protected by a European patent EP 1425993 [2].

Figure 3.
Rolf Benz testing armchair.

This armchair enables the setting of optimal seat furniture parameters. In selecting a sofa for multiple users (family, household) with a significantly different stature, it is necessary for all users to test the armchair in order to achieve an acceptable compromise.

ELECTRIC TESTING SOFA

Current adjustable seating furniture generally uses electric drives to increase the ease of operation and use. Mobile phone and tablet control brings technical and commercial attractiveness. The testing armchair equipped with electric drives enables comfortable and commercially attractive testing of optimal seating furniture parameters by multiple users at a time.

Seat height

By adjusting the seat height to the programmed positions it is possible for multiple users to comfortably test the optimal seat height.

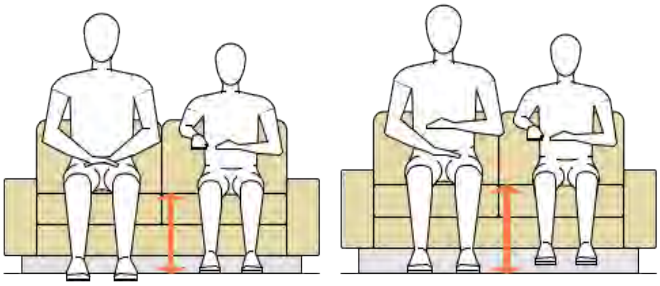

Fig. 4.
Optional seat height.

For example, the Linak Baselift [3] drive enables comfortable testing of seat height in the range of 40 ~ 50cm; with a linear drive for example Linak DL1A [4], a seat height in the range of 35 ~ 55 cm can be achieved.

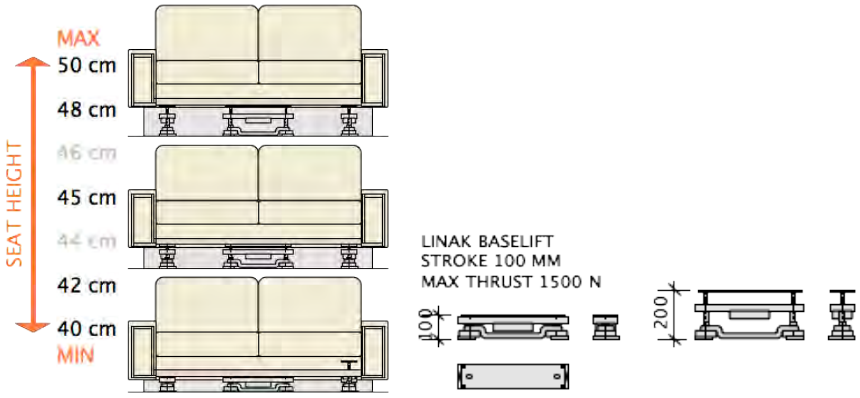

Fig. 5.
Optional seat heigh tin range of 40-50cm.

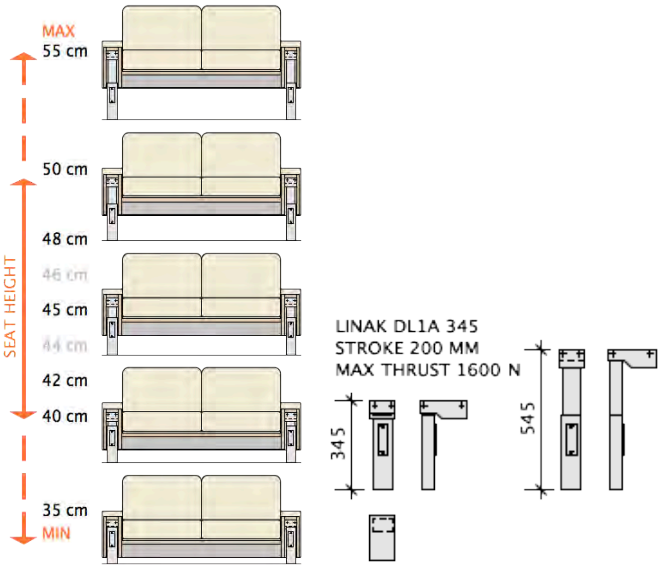

Fig. 6.
Optional seat heigh tin range of 45-55cm.

Armrest height

The height adjustable armrests enable comfortable testing and determination of armrest height above the seat.

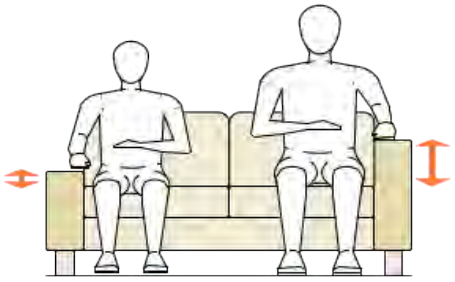

Fig. 7.
Optional armrest height.

Current mechanical mechanisms used mainly for backrest height adjustment are available.

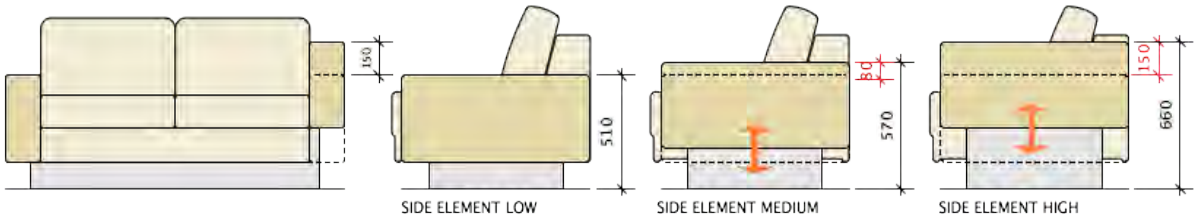

Fig. 8.
Mechanic adjustable armrest height.

A "growing" armrest with an adjustable height can be an innovative solution. The adjustment mechanism may be equipped with an electric drive. Elastic upholstery fabric with 100-130% expandability allows armrest height flexibility.

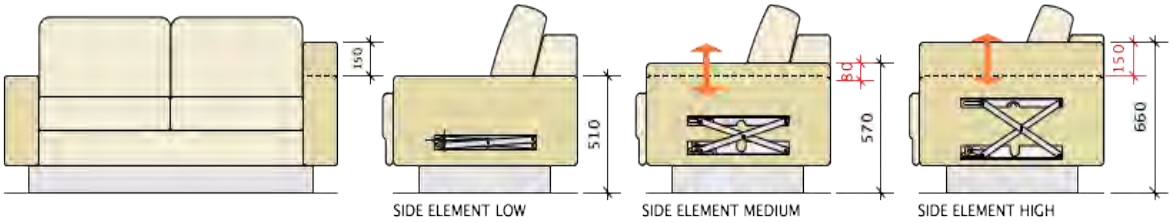

Fig. 9.
Electric adjustable armrest height.

If a figure is wider than a single column, it may be given as to cover two columns and it should be placed at the top or bottom of the page.

Armrest width

The armrest width is not a crucial ergonomic parameter of seating furniture, but it is another element that affects the overall seating comfort. The armrest width can be tested with a simple combination of wide and narrow armrests at the sides of the sofa.

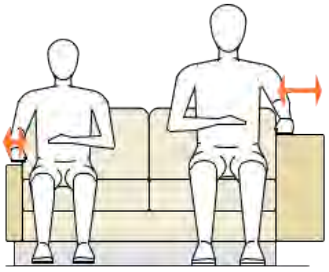

Fig. 10.
Optional armrest width.

If a presentation of more width variations is necessary in the effort to create a sophisticated complex testing sofa, technical adjustment of armrest width is possible.

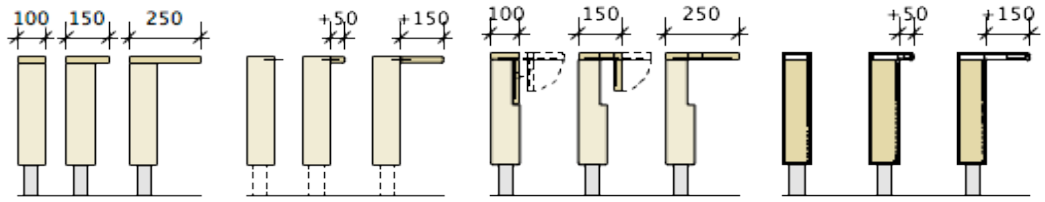

Fig. 11.
Examples of adjustable armrest width.

Seat depth

The testing sofa can be equipped with seats of varying depth. The optimal seat depth can be tested by sitting on each of these seats.

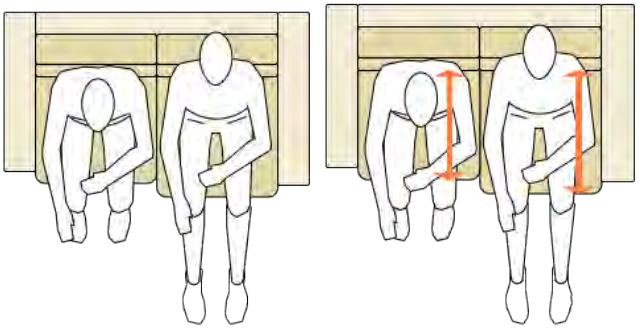

Fig. 12.
Optional seat depth.

Adjustable mechanisms, which are currently used in seating furniture to change the seat depth, can be used for comfortable testing of selectable seat height without having to switch between seats. Mechanical adjustment mechanisms are available; mechanisms with an electric drive offer the highest comfort.

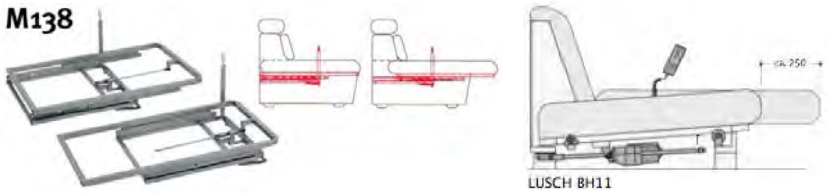

Fig. 13.
Mechanical mechanism Ipea M138 [5] and electric mechanism Lusch BH11 [6].

Seat hardness / softness

Cushion hardness testing (especially on the seat) is possible by sitting on seat samples of varying hardness or with a different cushion composition.

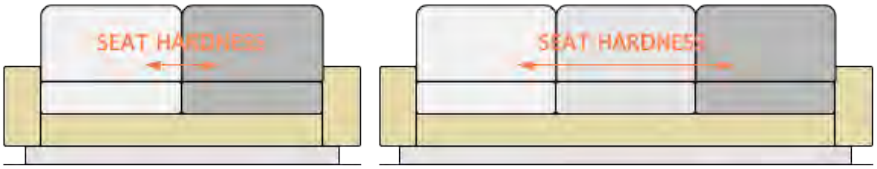

Fig. 14.
An example figure. For a larger range of selectable cushioning, the seats and backrests can be replaced.

Safety system

To ensure safety, the testing sofa with adjustable height can be equipped with safety features to prevent injury to persons or damage to objects. Safety contact strips placed in high risk areas are currently used for this purpose.

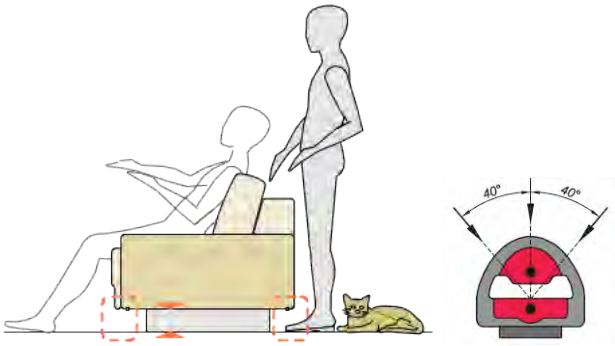

Fig. 15.
Danger zones with safety edge [7].

UNIVERSAL TESTING SOFA

The "square" shape of the testing sofa can accurately simulate most conventional models of contemporary seating furniture. Seating furniture models with unique specific shapes would optimally require adequate test sofa designs.

CONCLUSIONS

Testing sofas as a business tool enable easy, comfortable and dignified testing of selectable seating furniture size parameters. With the use of current mechanical and electric drives, it is possible to create suitable testing sofas according to business requirements of the manufacturer/distributor of the seating furniture. The testing process itself can be very attractive to customers and significantly strengthen their confidence in the optimal parameters of "their" seating furniture.

ACKNOWLEDGMENT

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COMPARATIVE STUDY ON THE THERMAL CONDUCTIVITY AND ACOUSTIC ABSORPTION OF THERMO- TREATED AND NON- TREATED ASH WOOD

Phd.eng. Sergiu Valeriu GEORGESCU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
Tel: 0040 268 419581, Fax: 0040 268 419581, E-mail: sergiu.georgescu@unitbv.ro

Prof.dr.eng. Camelia COŞEREANU
Transilvania University of Brasov, Faculty of Wood Engineering
Str. Universitatii nr. 1, 500068 Brasov, Romania
E-mail: cboieriu@unitbv.ro

Abstract

The paper presents the results of an experimental research performed with ash wood (Fraxinus Excelsior) in two variants: thermally treated and non-treated wood. Ash wood lamellas in the two variants were glued by vinyl polyacetate, forming boards tested to thermal conductivity and sampled further for acoustic absorption and density tests. The results showed that the thermal conductivity coefficient (λ) decreases in case of thermally treated ash wood in range 3,45% to 3,73%. By comparing the values obtained for the heat-treated vs. non-treated ash wood, it can be noticed that heat-treated wood panels are better thermal insulators than the non-treated ash wood panels. This is an advantage of using it for multi-layered structures of wooden houses, for outdoor applications. Instead, the thermally treated ash wood had a slightly lower acoustic absorption power compared with non-treated wood, requiring solutions for increasing sound insulation performances.

Key words: *thermal conductivity; ash wood; non-treated wood; thermally modified wood; acoustic absorption.*

INTRODUCTION

Ash wood is one of the wood species used for thermal treatment, in order to be used for outdoor applications. Thermal conductivity values were determined for ash wood (Şahin Kol 2009) in the range 0.113 to 0.202 W/mK. Thermal conductivity increased with increasing moisture content (MC) in the range of 0 to 22 percent. Radial thermal conductivity was 1.06 times tangential thermal conductivity for ash wood. In BS EN 12524 specified thermal conductivity (design value) to 0.13 W/mK and 0.18 W/mK for wood density of about 500 kg/m³ and about 800 kg m³, respectively in the conditions when thermal conductivity (λ) increases approximately linearly with increasing density and moisture content. The data bases (https://www.matbase.com/) show the density of 650 kg/m³ and thermal conductivity (λ) of 0.17 W/mK for ash wood.

Thermal modification of ash wood at a temperature of 180–220 °C causes the changes in wood characteristics, such as the colour darkens, increased dimensional stability, decreased water absorption, heat conductivity lowered up to 20-30%, wood density decreases (http://thermoarena.com/). The thermo-treated wood data basis (http://www.thermotreatedwood.com) indicates a moderate (OK) thermal conductivity of dark and medium thermo-treated ash wood compared with non-treated species of wood. More precise data can be found in the literature (Niemz et al 2010, Olărescu et al. 2015) for several species of wood. For thermo-treated ash wood at a temperature of 240°C, the values of 0,123 W/mK and 0,128 W/mK were experimentally determined for thermal conductivity (λ). Thus, a reduction of thermal conductivity in the range 6.5% to 10.2% was registered for thermal treated ash wood compared to non-treated wood. The experimental results on other species of wood, namely lime wood and spruce wood (Olărescu et al. 2015) show that the thermal conductivity decreases in the range 6% to 13% compared to non-treated wood.

Application fields of ash treated wood are decks, claddings, noise barriers, terrace floors, doors and windows, and musical instruments, too (Zauer and Pfriem 2010, Zauer et al. 2014). The acoustic properties of thermally modified maple (*Acer pseudoplatanus* L.) wood and beech (*Fagus sylvatica* L.) wood were investigated with good results in increasing the acoustic and mechanical properties similar to Hard maple. The results clearly shown that thermal treatment improves the sound quality of wood.

Wood is a light material, but alone is not a good absorption material. Wood conducts sound better in the longitudinal direction of the grain than perpendicular to it. A sufficient level of sound

insulation in wooden buildings can be achieved structurally only by using multi-layered constructions and positioning porous absorption material behind the panelling board. Thus, a so-called board resonator is formed which, when it vibrates, effectively dampens low sounds. On this line, the use of thermal treated wood for outdoor panelling could be a solution for such types of multi-layered structures, but knowledge of the thermal and sound insulation performances are required.

OBJECTIVE

The present paper's main objective is to investigate the thermal and sound insulation properties of treated and non-treated ash wood, as a prospective material uses for multi-layered structures designed for wooden houses, in outdoor conditions. For this purpose, thermal conductivity coefficient and sound absorption coefficient were experimentally determined for both treated and non-treated ash wood panels.

MATERIAL, METHOD, EQUIPMENT

The two types of panels used for the experiment were executed from thermal treated and non-treated ash wood lamellas. The thermally modified ash wood lamellas were bought from the thermowood market. The wood lamellas were glued by vinyl polyacetate, forming boards of 630mm x 630mm x 27mm for thermal conductivity test. The panels were cold pressed for 24 hours and conditioned for two weeks. The boards were afterwards sized at the final dimensions of 600mm x 600mm x 27mm and weight for calculating the density (Table 1).

Table 1

The characteristics of the experimental boards designed for thermal conductivity test

Panel code number	Panel sizes			Volume	Weight	Density
	Length [mm]	Width [mm]	Thickness [mm]	[m3]	[Kg]	[Kg/m3]
PFNT	600	600	27	0,00972	6,41	659,47
PFTT	600	600	27	0,00972	6,12	629,63

The specimens of 100mm diameter, required for the acoustic absorption coefficient determination were cut from panels with sizes of 600mm x 110mm x 27mm, obtained in the same way as described before.

The equipment used for measuring the thermal conductivity coefficient was HFM 436 Lambda heat flow meter (Fig. 1). The square sample of 600mm x 600mm is positioned between a hot and a cold plate. The stationary heat transfer through the sample material is measured by means of heat flow sensors embedded in the plates.

Fig. 1.
HFM 436 Lambda heat flow meter used for thermal conductivity determination

The heat flow passes the sample from the upper to the lower surface. The sensors selected for measurement are the central ones, located in an area of 250mm x 250mm. The measurements were carried out for temperature differences between the plates (ΔT) of 20°C and 30°C in eight measuring points according to the values shown in Table 2 and Table 3. Before the panels were measured, the equipment was calibrated to the ΔT value shown in the tables below. The measurements were carried out in accordance with the provisions of ISO 8301/1991.

Table 2

The measuring points for ΔT=20^{0}C

Measuring points	Temperature of cold plate T1	Temperature of hot plate T2	ΔT=T2-T1	Average (T2+T1)/2
	[^{0}C]	[^{0}C]	[^{0}C]	
1	-20	0	20	-10
2	-15	5	20	-5
3	-10	10	20	0
4	-5	15	20	5
5	**0**	**20**	**20**	**10**
6	5	25	20	15
7	10	30	20	20
8	15	35	20	25

Table 3

The measuring points for ΔT=30^{0}C

Measuring points	Temperature of cold plate T1	Temperature of hot plate T2	ΔT=T2-T1	Average (T2+T1)/2
	[^{0}C]	[^{0}C]	[^{0}C]	
1	-20	10	30	-5
2	-15	15	30	0
3	-10	20	30	5
4	-5	25	30	10
5	0	30	30	15
6	5	35	30	20
7	10	40	30	25
8	15	45	30	30

The samples used for the determination of sound absorption coefficient were circular shapes cut using a round pattern of 100 mm diameter (Fig. 2). The specimens were given code numbers as shown in Table 4. Five specimens each type were used for testing.

The specimens were tested using an impedance tube - Kundt tube (Fig. 3), on which two microphones have transmitted the measured data to a specialized software. In order to measure the sound absorption coefficient, the frequency ranged between 50 Hz and 1390 Hz and the sound level tested was of 75 dB.

The maximum values of the sound absorption coefficients were calculated by the software of the equipment.

Fig. 2.
The samples subjected to sound absorption testing.

Table 4

Code numbers of the specimens used for sound absorption testing

Code number	Type of ash wood	Number of specimen
FT1	Thermo-treated wood	**1**
FT2	Thermo-treated wood	**2**
FT3	Thermo-treated wood	**3**
FT4	Thermo-treated wood	**4**
FT5	Thermo-treated wood	**5**
FN1	Non-treated wood	**1**
FN2	Non-treated wood	**2**
FN3	Non-treated wood	**3**
FN4	Non-treated wood	**4**
FN5	Non-treated wood	**5**

Fig. 3.
The impedance tube used to measure the sound absorption coefficient.

The samples used for the determination of density profile were cut at sizes of 50mm x 50mm. The test was carried out on X-ray density profile analyser DPX300, IMAL production (Fig. 4). Eight specimens of each type of investigated material (heat treated and non-treated wood) were tested. The DPX300 is used to supply the density profile of solid wood or wooden based panels, measured along the thickness. Based on the X-ray control, the equipment can analyse the density profile without any contact between material and measuring instrument. It comprises an X-ray source and a receiver, between which the density profile sample is placed.

Fig. 4.
X-ray density profile analyser DPX300, IMAL production.

RESULTS AND DISCUSSION

The thermal conductivity coefficients experimentally determined are presented in Table 5 for a temperature difference between the plates ΔT = 20°C and in Table 6 for a temperature difference between the plates ΔT = 30°C.

The obtained results show that the thermal conductivity coefficient (λ) decreases in case of thermally treated ash wood, with a mean value of 3,73% for a temperature difference between the plates ΔT = 20°C and a mean value of 3,45% for a temperature difference between the plates ΔT = 30°C. By comparing the values obtained for the heat-treated vs. non-treated panels, one can notice that the heat-treated wood panels are better thermal insulators than the non-treated ash wood panels. This is an advantage of using it for multi-layered structures of wooden houses, for outdoor applications.

The research results found in the literature (Şahin Kol 2009, Niemz et al. 2010, Olărescu et al. 2015) have shown that the thermal conductivity values increase with the density of material.

Table 5

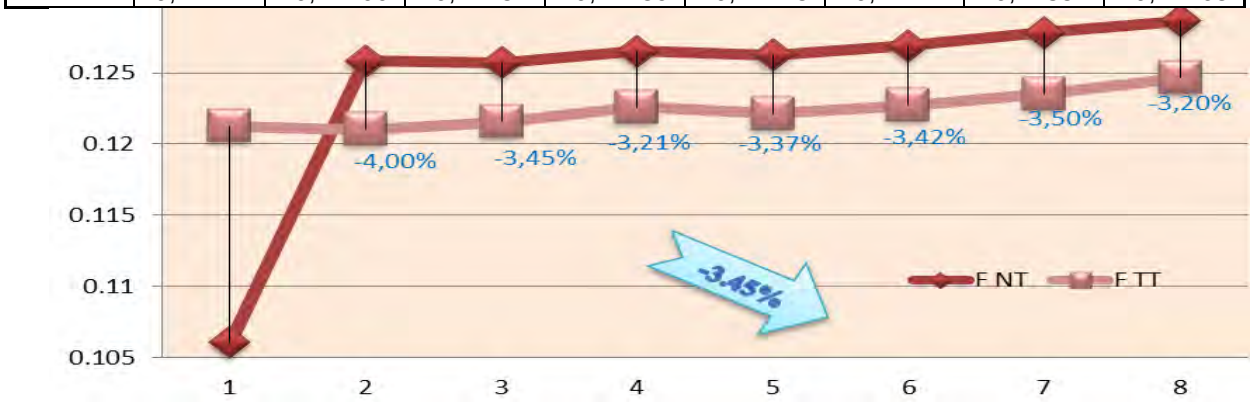
Thermal conductivity coefficient of the tested specimens [W/mK] la ΔT=20°C

Sample code no.	The measuring points							
	1	2	3	4	5	6	7	8
F NT	0,12578	0,12530	0,12693	0,12637	0,12593	0,12591	0,12671	0,12867
F TT	0,11966	0,12053	0,12178	0,12208	0,12193	0,12206	0,12272	0,12447

Table 6

Thermal conductivity coefficient (λ) of the tested specimens [W/mK] la ΔT=30°C

Sample code no	The measuring points							
	1	2	3	4	5	6	7	8
F NT	0,10608	0,12584	0,12573	0,12653	0,12627	0,12692	0,12784	0,12864
F TT	0,12124	0,12100	0,12154	0,12259	0,12215	0,12272	0,12352	0,12465



Determination of density values and density profile was carried out on specimens of 50m x 50m. X-ray density profile analyser DPX300, IMAL production has been used as equipment.

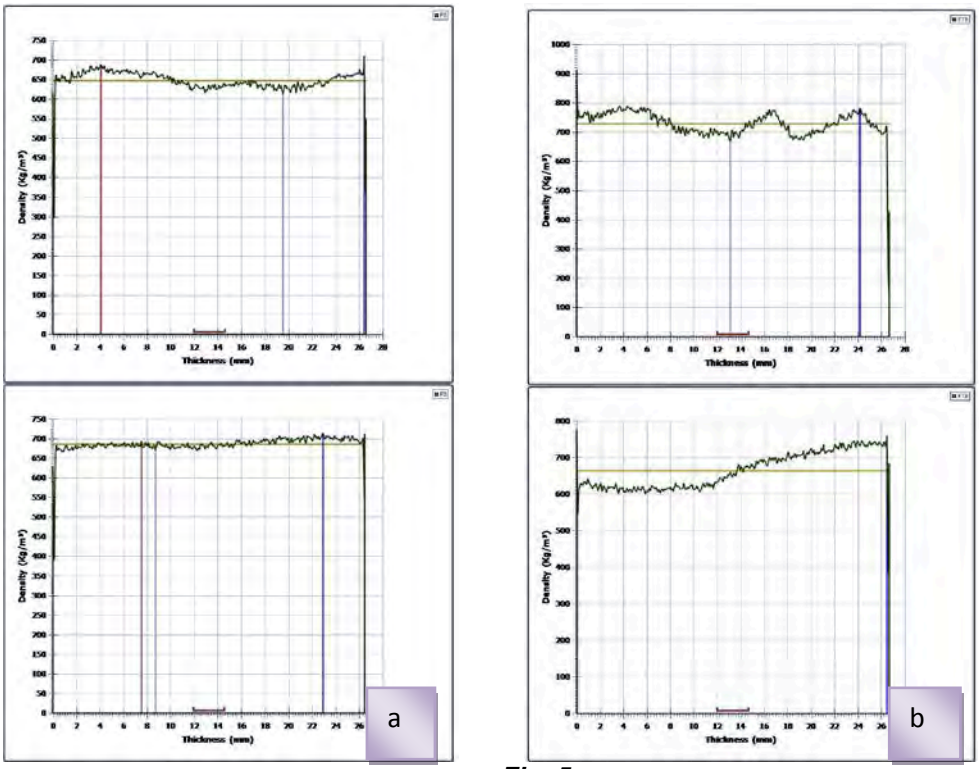


Fig. 5.
Density profile of non-treated wood (a) and thermally treated wood (b).

The mean values of densities obtained for non-treated wood and for heat treated wood were 682kg/m^3 and 632kg/m^3 respectively. A density reduction of 8% was registered for heat treated wood compared with non-treated wood. Four examples of density profiles are presented in Fig. 5. As noticed, the density profile of thermally treated wood has more irregularities compared with that of non-treated wood. It is explained by chemical modification of wood due to heating treatment and also by changes of microscopically structure.

The results of acoustic absorption of the specimens made from heat-treated and non-treated ash wood (Fig. 6 and Fig. 7) proved a low sound absorption of these materials for frequencies higher than 100Hz. Low frequencies sounds of 50 Hz have a higher absorption rate in the range 0,5 to 0,7. High and medium frequencies has poor sound absorption rate, around 0,1. The result confirms the necessity of using wooden board in multi-layer structures with porous materials as cores, so to obtain a satisfactory sound dumping. Yet, non-treated ash wood seems to be a better sound insulation material compared with treated one. The explanation could be the higher air content of non-treated wood stored in wooden cells.

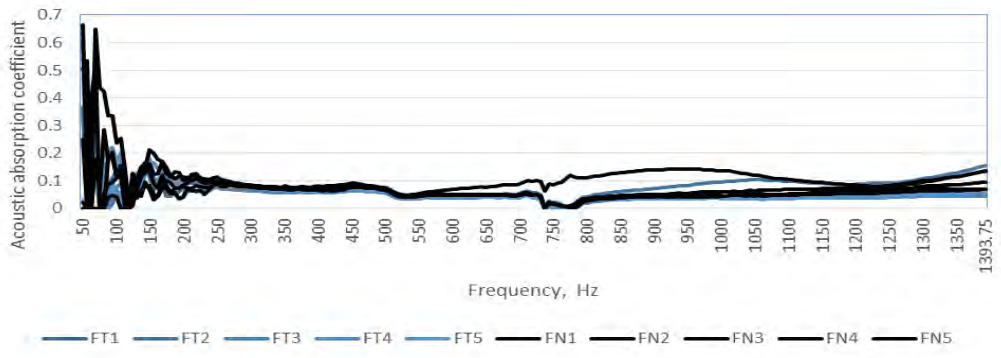


Fig. 6.
Acoustic absorption coefficient of treated ash wood samples (FT1...FT5) and non-treated ash wood samples (FN1....FN5).

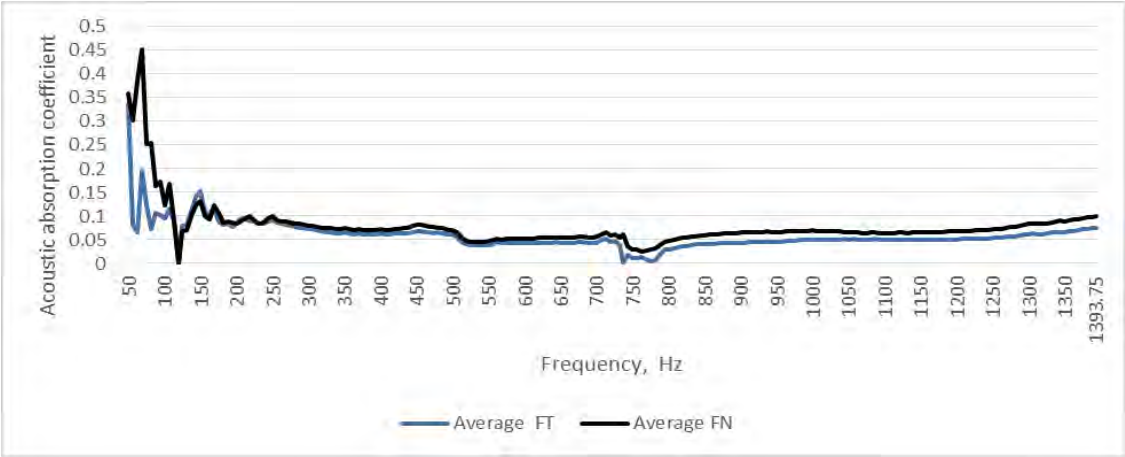


Fig. 7.
Mean values of acoustic absorption coefficient of treated ash wood (Average FT) and non-treated ash wood (Average FN).

CONCLUSIONS

The results obtained within the present research demonstrates that heat-treating the ash wood may increase the thermal insulation properties of this material, proved by the reduced values of thermal conductivity coefficients, with approx. 3%. Contrariwise, the thermally treated ash wood has a slightly lower acoustic absorption power compared with non-treated wood, fact that recommends it in multi-layer structures for an increased sound insulation performance.

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FIBER MORPHOLOGY OF ORANGE WOOD (*Citrus X sinensis* (L.) Osbeck)

Mehmet AKCA
Balıkesir University, Dursunbey Vocational School, Forestry and Forest Products Program
Balıkesir University Çağış Campus (Bigadiç over path 17. km) 10145, Balıkesir, Turkey
Tel: +90 266 612 14 00 – 08, E-mail: makca@balikesir.edu.tr

Hacı Ismail KESIK
Kastamonu University, Faculty of Forestry, Department of Forest Industrial Engineering
Kastamonu University Kuzeykent Campus 37150, Kastamonu, Turkey
Tel: +90 366 280 17 05, Email: hismailkesik@kastamonu.edu.tr

Alperen KAYMAKCI
Kastamonu University, Faculty of Forestry, Department of Forest Industrial Engineering
Kastamonu University Kuzeykent Campus 37150, Kastamonu, Turkey
Tel: +90 366 280 17 05, Email: akaymakci@kastamonu.edu.tr

Abstract

This study was to investigate some of the anatomical properties of orange (Citrus X sinensis (L.) Osbeck) wood naturally grown in Turkey. For this purpose, samples from cut trees were taken according to TAPPI T 257 cm-85 standards. The specimens were disintegrated in the size of a matchstick and specimens were prepared before the experiment. To measure the anatomical characteristics, Orange Tree woods (after removing barks) were macerated in a solution containing 1:1 HNO_3 and $KClO_3$. For maceration taken wood samples from three parts of each Orange Tree wood were chosen. A drop of macerated sample was taken on a slide and fiber length, fiber width, lumen width and cell wall thickness were measured under a microscope. According to the obtained data, the fiber length of the orange tree was found to be lower than that of the other broad-leaved tree species.

Key words: *orange wood; fiber length; lumen; cell wall.*

INTRODUCTION

Wood is one of the most widely used materials not only in wood processing industry, but also in other fields of industry (construction, chemistry, machinery, etc). Wood as material is very advantageous: it is easily processed (compared to metals and stone), strength of resistance, hardly affected by acids and alkali, has low heat conductivity, characterized by good adhesion properties, pleasant appearance and good finishing. Besides there are some disadvantages of wood. its strength, hardness and other mechanical properties differ in different directions; usually wood has defects, which worsen the quality of wood and its product (Fridley et al. 1996; McDonald et al. 1996).

Sweet orange (Citrus sinensis L. Osbeck) commonly called orange is a member of this family and a major source of vitamins, especially vitamin C, sufficient amount of folacin, calcium, potassium, thiamine, niacin and magnesium (Angew 2007). Economically, oranges are important fruit crops, with an estimated 60 million metric tonnes produced worldwide as at 2005 for a total value of 9 billion dollars. Of this total, half came from Brazil and the United States of America (Goudeau et al. 2008; Bernardi et al. 2010). This study was to investigate some of the anatomical properties of orange (Citrus X sinensis (L.) Osbeck) wood naturally grown in Turkey.

MATERIALS AND METHOD

Materials

Three orange trees (*Citrus X sinensis (L.)* Osbeck) were used as experimental material in Tarsus province of Mersin. Samples from cut trees were taken according to TAPPI T 257 cm-85 standards. The specimens were disintegrated in the size of a matchstick and specimens were prepared before the experiment.

Methods

The wood specimens of (*Citrus X sinensis (L.)* Osbeck) were extracted from branch of the trunks. For preparation of wood sections and macerations, standard procedures were applied (Yaltirik 1971).

To measure the anatomical characteristics, Orange Tree woods (after removing barks) were macerated in a solution containing 1:1 HNO_3 and $KClO_3$. For maceration taken wood samples from three parts of each Orange Tree wood were chosen. A drop of macerated sample was taken on a slide and fiber length, fiber width, lumen width and cell wall thickness were measured under a microscope. For measuring fiber length and diameter, 100 fibers were measured and average reading was taken.

Olympus CX21 light microscope with micrometer graduated ocular was used for fiber measurements. The fiber length is measured in the X10 objective, the fiber width, the lumen width and the double wall thickness measured in the X40 objective. The criteria used in the evaluation of fiber, board and paper industry are explained below.

Elasticity coefficient (%): (Lumen diameter ÷ Fiber diameter)×100
Felting rate: Fiber length ÷ Fiber diameter
Runkel index: Cell wall thickness ÷ Lumen Diameter
Rigidity coefficient (%): (Cell wall thickness ÷ Fiber diameter)×100
F ratio (%): (Fiber length ÷ Cell wall thickness) ×100
Muhlstep classification: (Cell wall area ÷ fiber cross-sectional area) ×100

RESULTS AND DISCUSSION

Fiber morphology values of orange wood are given in Table 1. Fiber cells image is shown in Fig. 2.

Table 1

Some statistics fiber dimensions of orange wood

***Citrus X sinensis (L.) Osbeck* (Orange Tree wood)**				
Variables	**Fiber length (mm)**	**Fiber width (μm)**	**Lumen width (μm)**	**Single cell wall thickness (μm)**
Total Data	100	100	100	100
Average	0.54	36.26	18.78	8.74
Max.	0.79	50	31	14.5
Min.	0.27	18	9	4
Standart Deviation	0.11	5.98	4.52	2.31
Standart Error	0.1	0.60	0.45	0.23
Variance Coefficient	20.08	16.49	24.05	26.37

According to Table 1, the fiber length of the orange tree is 0.54mm, the fiber width is 36.26μm, the lumen width is 18.78μm and the single cell wall thickness is 8.74μm.

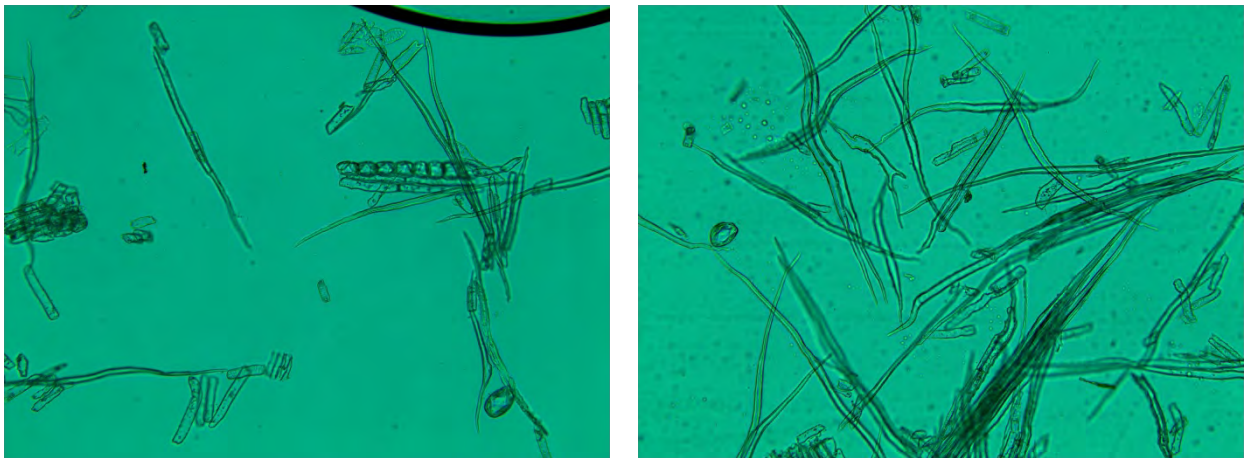

Fig. 2.
Fiber images.

Some data (Elasticity Ratio, felting ratio, Runkel classification, Rigidity coefficient, Muhlstep classification, F factor) calculated by proportioning the fiber sizes of the orange tree to each other are given in table 2.

Table 2

Values belong to morphological properties of orange tree

Species	Elasticity ratio	Felting ratio	Runkel classification	Rigidity coefficient	Muhlstep classification	F factor
Orange Tree	51.79	14.89	0.93	24.1	26.82	308.92

The morphological characteristics of the orange tree were found to be elasticity coefficient 51.79, felting ratio 14.89, runkel classification 0.93, rigidity coefficient 48.2, muhlstep classification 26.82, F factor 308.92.

Comparison of the fiber size and morphological properties of the orange tree with some leaf tree data is given in Tables 3 and 4.

Table 3

Comparison of fiber sizes of orange tree with some broad-leaved tree

Variables	Fiber length (mm)	Fiber width (mm)	Lumen width (mm)	Single cell wall thickness (µm)	References
Citrus X sinensis (L.) Osbeck	0.54	36.26	18.78	8.74	Current Study
Populus tremula	1.08	26.87	14.75	6.06	Alkan et al. 2003
Populus nigra	1.25	27.17	17.70	4.98	Alkan et al. 2003
Salix alba	1.19	24.10	16.10	4.00	Alkan et al. 2003
Fagus orientalis	1.08	19.50	4.75	7.37	Alkan et al. 2003
Quercus robur	1.09	20.17	10.55	4.81	Alkan et al. 2003
Quercus petraea	1.13	20.65	7.20	6.72	Alkan et al. 2003
Castanea sativa	1.06	21.15	11.60	4.77	Alkan et al. 2003
Carpinus betulus	1.23	20.22	9.20	5.51	Alkan et al. 2003
Fraxinus excelsior	1.07	21.20	10.37	5.41	Alkan et al. 2003
Acer campestre	0.73	21.17	13.45	3.86	Alkan et al. 2003
Juglans regia	1,46	22,82	13,22	4,80	Alkan et al. 2003
Platanus orientalis	1,47	27,72	9,95	8,88	Alkan et al. 2003

Table 4

Comparison of the morphological characteristics of orange tree with some broad-leaved tree

Variables	Elasticity ratio	Felting ratio	Runkel classification	Rigidity coefficient	Muhlstep classification	F factor	References
Citrus X sinensis (L.) Osbeck	51,79	14,89	0,93	24,1	26,82	308,92	Current Study
Populus tremula	54,89	40,04	0,82	22,55	69,86	177,50	Alkan et al. 2003
Populus nigra	65,14	45,96	0,56	18,32	57,56	250,75	Alkan et al. 2003
Salix alba	66,80	49,53	0,49	16,59	55,37	298,45	Alkan et al. 2003
Fagus orientalis	24,35	55,60	3,10	37,79	94,06	147,11	Alkan et al. 2003
Quercus robur	52,30	54,14	0,91	23,84	72,64	227,05	Alkan et al. 2003
Quercus petraea	34,86	54,53	1,86	32,54	87,84	167,58	Alkan et al. 2003
Castanea sativa	54,84	50,18	0,82	22,55	69,91	222,51	Alkan et al. 2003
Carpinus betulus	45,49	60,97	1,19	27,25	79,29	223,74	Alkan et al. 2003
Fraxinus excelsior	48,91	50,61	1,04	25,51	76,07	198,33	Alkan et al. 2003
Acer campestre	63,53	34,50	0,57	18,23	59,63	189,23	Alkan et al. 2003
Juglans regia	57,93	63,87	0,72	21,03	66,43	303,68	Alkan et al. 2003
Platanus orientalis	35,89	53,12	1,78	32,03	87,11	165,82	Alkan et al. 2003

As the fiber length of the wood material is affected by the tearing resistance of the paper and as the fiber length increases, the tear resistance increases with the interfiber adhesion surface increase (Dadswell ve Watson 1962). According to the obtained data, the fiber length of the orange tree was found to be lower than that of the other broad-leaved tree species in table 3.

The elasticity ratio of the orange tree is similar to that of the other broad-leaved tree with 51.79% in Table 4 and the elasticity ratio of fibers between 50-75% is considered elastic (Alkan et al. 2003). The ratio of fiber length to fiber width gives us a felting ratio. This ratio gives us an idea about resistance to tearing from physical resistance properties. The rate of felting in the orange tree is found 14.89% due to the short fiber length. The high coefficient of rigidity has a negative effect on the physical resistance of the paper (Akkayan 1983; Göksel 1986). The orange tree rigidity coefficient was found to be 24.1% which is much lower than that of many broad-leaved tree in Table 4. The orange tree Mühlsteph classification was found to be 24.1% which is much lower than that of many broad-leaved tree in Table 4. F factor gives us information about the flexibility of the paper to be produced (Casey 1961). As the F factor increases the flexibility of papers increases (Casey 1961). The F factor (308.92%) in the orange tree was found to be significantly higher than the other broad-leaved tree species except for Juglans Regia (308.68) in Table 4.

CONCLUSIONS

This study was to investigate some anatomical properties of range wood naturally grown in Turkey. According to the obtained data, the fiber length of the orange tree was found to be lower than that of the other broad-leaved tree species. The rate of felting in the orange tree is found 14.89% due to the short fiber length. The high coefficient of rigidity has a negative effect on the physical resistance of the paper. F factor gives us information about the flexibility of the paper to be produced (Casey 1961). As the F factor increases the flexibility of papers increases (Casey 1961). The F factor (308.92%) in the orange tree was found to be significantly higher than the other broad-leaved tree species except for Juglans Regia (308.68).

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DENSITY AND COMPRESSION STRENGTH OF SCOTS PINE WOOD IN RELATION TO SITE AND SILVICULTURAL MEASURES

Ondřej SCHÖNFELDER
Czech University of Life Sciences Prague, Faculty of Forestry and Wood Sciences,
Department of Wood Processing
Kamycka 129, 165 21 Prague, Czech Republic
Tel: 00420 224 383 742, E-mail: zeidler@fld.czu.cz

Aleš ZEIDLER
Czech University of Life Sciences Prague, Faculty of Forestry and Wood Sciences,
Department of Wood Processing
Kamycka 129, 165 21 Prague, Czech Republic
Tel: 00420 224 383 742, E-mail: zeidler@fld.czu.cz

Vlastimil BORŮVKA
Czech University of Life Sciences Prague, Faculty of Forestry and Wood Sciences,
Department of Wood Processing
Kamycka 129, 165 21 Prague, Czech Republic
Tel: 00420 224 383 742, E-mail: zeidler@fld.czu.cz

Abstract

The paper presents the results of an experimental research performed with Scots pine wood (Pinus sylvestris L.) coming from different areas in the Czech Republic – Doksy region and Plasy region. Although pine is the second most important commercial softwood species in the Czech Republic, we know very little about factors influencing its wood properties. We chose two different regions and subsequently forest stands with different history of silvicultural management inside these areas. Sample trees were cut to evaluate wood density and compression strength following Czech national standards. Besides site conditions, the silvicultural measures and position within a stem were evaluated factors influencing the tested properties. The biggest density value of 0.488 g.cm^{-3} was obtained in a stand that was regenerated using the shelterwood method with long regeneration period in Doksy region, and the lowest density value of 0.451 g.cm^{-3} was obtained in a stand that was regenerated using the clear-cutting method in Plasy region. The biggest value of 49.3 MPa for compression strength was achieved in Doksy region in the stand that was regenerated using the shelterwood method. On the contrary, the lowest value of compression strength of 43.1 MPa was achieved in Plasy region in a stand that was managed by the clear-cutting method. A growing trend for wood density and compression strength was confirmed in direction from the stem centre to the bark. It could be concluded that silvicultural measures, site conditions and within-stem position are important factors influencing quality of pine wood.

Key words: *scots pine; wood; density; compression strength; variability; silvicultural measures; Czech Republic.*

INTRODUCTION

Scots pine (*Pinus sylvestris L.*) currently occupies more than 16 % of the total forest area in the Czech Republic and it so represents the second most important commercial softwood species in the country. It is presupposed that its importance will be increasing in the near future as Norway spruce (*Picea abies* (L.) H. Karst), the most important commercial species, has been fading because of climatic changes. The state forest policy is considering the idea to increase the share of pine in forest stands currently (MZe 2016).

Wood quality is in focus of both foresters and wood processing industry. It has been discussed a lot and it is a topic of many research works. It must be understood in the relevant context, usually from the view of properties that are important for the processing and utilisation of the wood. Wood density is a kind of characteristic that is widely used for wood quality evaluation (Jozsa, Middleton 1994). The reason the wood density could be used as an indicator of wood quality is a fact that it influences other physical, and especially mechanical properties of wood to great extent (Auty et al. 2014; Kollmann 1951). It generally applies that with increasing density the strength of wood also increases (Tsoumis 1991).

Wood is actually a result of growing processes in a living tree and exhibits so high variability of properties. The variability of properties can be found among individual regions or trees as a result of

genotype. Especially the variability of wood properties within a trunk could be critical for the final wood processing. Wood properties are influenced to great extent by climate or altitude, silvicultural practises also play role (Tomczak et al. 2007; Kask 2015; Tsoumis 1991; Peltola et al. 2007).

A certain pattern can be found for wood density variability within a stem. It increases in the radial direction from the pith to the bark of the trunk (Požgaj et al. 1997). It is presumed that occurrence of juvenile wood in the central part of the stem is the mail reason for this variability. Another important factor that influences variability of density in the horizontal direction is the width of the annual rings. It is assumed for softwoods that with increasing ring width, the proportion of latewood decreases, and consequently its density also decreases (Kask 2015; Tsoumis 1991).

OBJECTIVE

The main objective of our research was to evaluate the variability of oven-dry density and compression strength along the fibres of Scots pine grown at different regions in the Czech Republic. As the mail source of variability, the site conditions and silvicultural measures were tested. The impact of position within a stem in horizontal direction was also evaluated.

MATERIAL, METHOD, EQUIPMENT

The wooden material presented in this study comes from two different regions in the Czech Republic. The first one called Doksy region, situated in the north part of the Czech Republic, with average precipitations of 550 mm, and the average temperature is between 7-8 °C. The altitude reaches up to 450 m above sea level. The second region is called Plasy, situated in south-western part of the country, with average precipitations between 500 – 550 mm. The altitude reaches between 500 – 620 m above sea level. Both of the regions are representative for growth of pine in the Czech Republic. In each region we chose always two forest stands with different silvicultural history. The first one had been managed with shelterwood method and released after a certain time. The second forest stand has been managed with clear-cutting method. More details about the stands and trees in them is presented in Table 1. In each forest stand we cut seven sample trees. The sample trees were representative for the individual forest stand and free of any defects and irregularities.

Table 1

Desription of forest stands

		Age* *(years)*	SLT**	Silvicultural practice	Stem diameter (mm)	Tree height (m)
Doksy region	Stand 1	25	0K4	Shelterood	169	13.0
	Stand 2	49	0K4	Clear-cutting	187	18.1
Plasy region	Stand 1	36	4Q1	Shelterood	160	14.1
	Stand 2	25	4Q1	Clear-cutting	180	17.1

**according to forest management plan*

***set of forest types according to the Czech typological system (Viewegh et al. 2003)*

Totally 28 sample trees were cut in the selected stands to evaluate physical and mechanical properties of pine wood. A section (150 cm long) was cut off from basal area of the each sample tree. A disc was also cut from the breast-height diameter area from the each sample tree to evaluate annual rings width (Fig. 1).

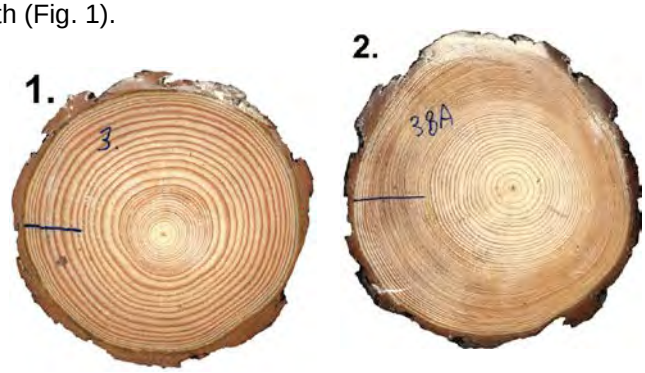

Fig. 1.
Disks from the sample trees – (1) shelterwood system of management (2) clear-cutting system of management.

A central board was removed from each section in order to assess the variability of the tested properties in the horizontal plane of the stem. After moisture content dropped below 15 %, the boards were cut lengthwise in the direction away from the pith toward the cambium and finally testing samples 20×20×30 mm (radial × tangential × longitudinal) for physical and mechanical test were prepared (see Figure 2).

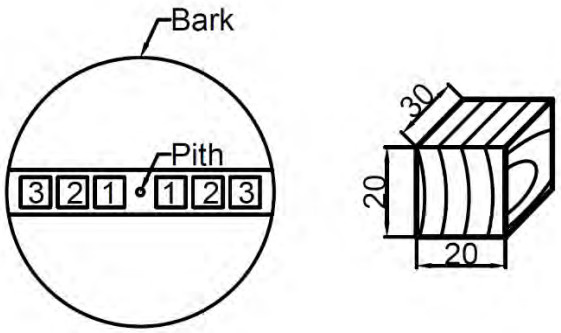

Fig. 2.
Tree sampling and the testing sample description

We examined first density as the basic variable. We tested oven-dry density (0 % MC of wood). Totally 866 testing samples were used to evaluate density. All the test were performed in compliance with Czech national standard (ČSN 49 0108). Oven-dry density was computed according to following formula:

$$\rho_0 = \frac{m_0}{V_0} \ [g.cm^{-3}],$$

where:
m_0 is the dry mass of the specimens $[g]$,
V_0 is the volume of dry specimens $[cm^3]$.

Compression strength along the fibres was tested according to Czech national standard (ČSN 49 0110). Totally 451 testing samples were used to evaluate compression strength. The property was computed using following formula:

$$\sigma_{12} = \frac{F_{12}}{a_{12}.b_{12}} \ [MPa],$$

F_{max} is the maximum load (N) at 12% MC
a_{12} and b_{12} are the transverse dimensions of the sample (mm) at 12% MC

The multifactor ANOVA tests (Fisher F – test) and Duncan's multiple comparison tests were employed to evaluate the impact of individual factors, especially silvicultural practises, site and the position from the pith. The level of significance α = 0.05 % was used for all statistical analyses. STATISTICA 12 software (Statsoft Inc., USA) was used to carry out all statistical analyses.

RESULTS AND DISCUSSION

Density

There is statistically significant difference between the regions. The biggest value of density was obtained for Doksy region. This fact, that site plays important role in wood density variability, is mentioned by Tsoumis (1991). It was also confirmed by many authors (Jelonek et al. 2005; Hautamäki et al. 2014; Tomczak and Jelonek 2013).

Figures of the descriptive statistics for wood density in relation to individual stand and regions are shown in Table 2. It is evident that density doesn't differ depending on silvicultural practices in the both regions. Most of the authors mention lower density for planted pines (Eriksson et al. 2006; Mederski et al.2015). Greater values were obtained for both stands from Doksy region. Nevertheless all obtained results for density are similar to those reported by Wagenführ (2002) for pine. Other

authors even mention lower values than the figures attained in our research (Tsoumis 1991; Požgaj et al.1997; Novák 1970).

Table 2

Descriptive statistics – comparison of density in individual stands

Density	Doksy region		Plasy region	
	Stand 1	Stand 2	Stand *1*	Stand *2*
Mean (g.cm^{-3})	0.488	0.487	0.455	0.451
Median (g.cm^{-3})	0.488	0.483	0.453	0.445
Coefficient of variation (%)	8.1	10.7	7.97	12.92
Standard deviation (g.cm^{-3})	0.039	0.052	0.036	0.058
Number of specimens (pieces)	173	284	160	249

Table 3

Descriptive statistics – within-stem density variability

		Horizontal variability		
	Density (g.cm^{-3})	First position	Second position	Third position
Doksy region	Stand 1	0.478	0.499	0.477
	Stand 2	0.458	0.491	0.551
Plasy region	Stand 1	0.438	0.468	0.477
	Stand 2	0.421	0.472	0.461

The variability of density in the radial direction is presented in Figure 3 and Table 3 with the average density values in individual positions. The density variability in horizontal direction for stands 2 (clear-cutting method of management) demonstrated a clear trend in increasing the value in the direction from the pith to the bark. Similar results were obtained by Ivković et al. (2013), Nicholls and Brown (1973) and Fritts et al. (1991). A different trend was found for forest stands managed by shelterwood method (stands 1). The value of density close to the pith is bigger compared to stands 2 and the increase of density is replaced by moderate decrease (Fig. 3). Similar pattern was reported by Eriksson et al. (2006). The variability of density within-stem in the horizontal direction for individual stands was also confirmed by a tree ring analysis (see Fig. 4).

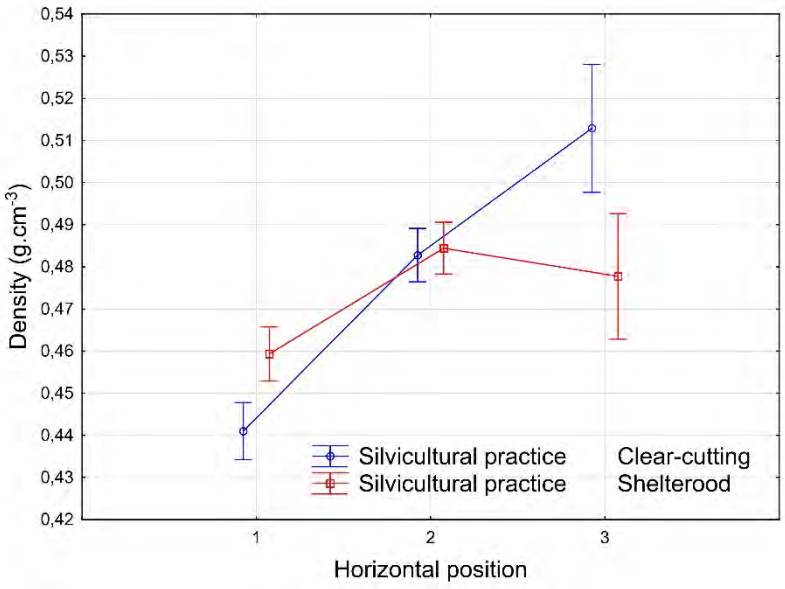

Fig. 3.
Within-stem variability of density in the horizontal direction.

Figure 4 presents results for the annual rings width analysis. It is clear from the chart that forest stands managed by the clear-cutting method exhibit wider annual rings close to the pith. With growing distance from the centre of the tree the rings are getting narrower and after certain time their width has stabilised. The same results obtained Tomczak et al. (2007). Stands managed by the shelterwood method exhibit completely opposite trend. This trend is also reflected in density variability in the horizontal direction, where density declines with increasing annual ring width. The same pattern were concluded by Gryc et al. (2011), Mörling (2002) and Ivković et al. (2013).

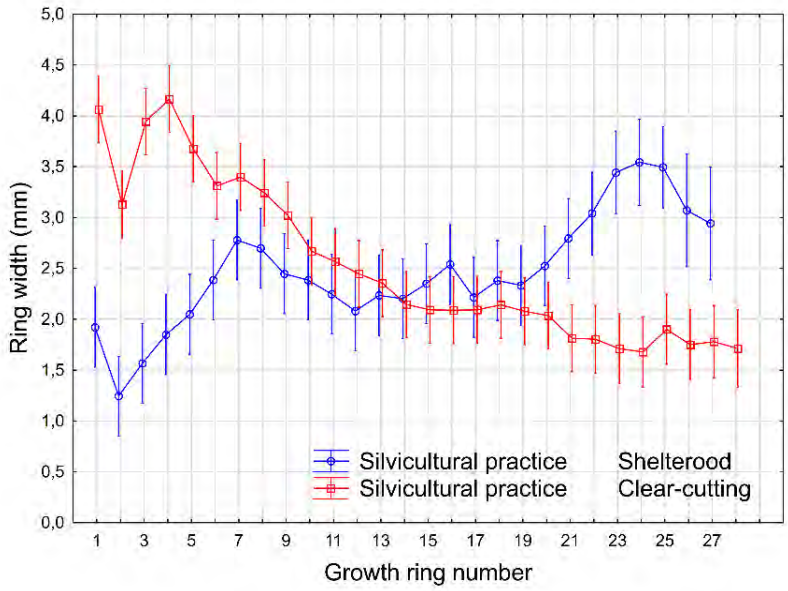

Fig. 4.
Impact of shelterwood method and clear-cutting method on annual rings width

Compression strength

The statistically significant difference between the two regions was confirmed for compression strength. The biggest value was obtained for Doksy region. This result is closely connected to the value of density, which was also bigger for this region. Požgaj et al. (1997) mention considerable impact of density on mechanical properties of wood. Different values for compression strength for different regions were obtained by Aleinikovas and Grigaliunas (2006). Figures of a descriptive statistics for compression strength in relation to individual stands and regions are shown in Table 4.

Table 4

Descriptive statistics – comparison of compression strength in individual stands

Compression strength	Doksy region		Plasy region	
	Stand 1	Stand 2	Stand 1	Stand 2
Mean (MPa)	49.3	46.2	43.1	45.8
Median (MPa)	49.3	44.9	43.5	44.8
Coefficient of variation (%)	11.6	22.0	16.5	18.1
Standard deviation (MPa)	5.7	10.1	7.1	8.3
Number of specimens (pieces)	96	132	77	146

The statistically significant difference among values of compression strength for individual stands was confirmed for the both regions. Table 4 shows average values of compression strength for the individual stands. The stands managed by shelterwood method reached bigger value in Doksy region, in contrast to Plasy region where bigger value was obtained for the stand managed by clear-cutting method. If compared to other authors (Požgaj et al. 1997; Witomski et al. 2014), the value of compression strength is lower irrespective to the stands. As far as the horizontal variability is a concern, the pattern similar to density fluctuation was confirmed for compression strength (Table 5).

Table 5

Descriptive statistics – within-stem compression strength variability

		Horizontal variability		
	Compression strength (MPa)	First position	Second position	Third position
Doksy region	Stand 1	48.3	50.8	45.3
	Stand 2	40.2	52.2	62.0
Plasy region	Stand 1	39.4	46.6	47.5
	Stand 2	41.1	50.1	47.2

CONCLUSIONS

The aim of this study was to evaluate the impact of silvicultural measures and site conditions on the wood density and the compression strength of Scots pine (*Pinus sylvestris*) from two different regions in the Czech Republic. In most cases, the evaluated wood density from the both regions achieves higher density values when compared to other authors. The compression strength along fibres achieved lower values when compared to other authors. Site conditions turned out to be significant factor influencing density and compression strength of pine wood. The impact of silvicultural methods was not confirmed for wood density. In the case of compression strength the silvicultural measures play a role. Shelterwood method provides wood with lesser variability of density and compression strength along stem radii.

ACKNOWLEDGEMENT

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THE IMPORTANCE OF INDUSTRIAL FOREST IN CELLULOSE AND PAPER PRODUCTION

Öznur ÖZDEN
Istanbul University, Forest Faculty, Department of Forest Products Chemistry and Technology
Address: 34473 Bahçeköy/İstanbul-Turkey
Tel: +90 212 3382400/25376, Fax: +90 212 3382424, E-mail: ozdeno@istanbul.edu.tr

Abstract

Continuity of cellulose and paper industry, which is one of the five biggest industries of the world, depends on the sustainability of raw material sources. The main raw material source for cellulose and paper production is perennial and annual plants. Annual plants alone are never enough for cellulose production. This is valid for both quality and contiunity. Sustainability should be taken into consideration and be accepted as a government policy for the use of appropriate perennial tree species in cellulose production.

Countries that produce cellulose generally use industrial forests and planned plantation. Although studies on industrial forests have been conducted for a while in Turkey, there has not been a considerable progress so far. The fact that cellulose is produced in only one factory is also one of the reasons of this.

In this study, the importance of industrial forests, the reason why they are important in cellulose production and the current situation in Turkey has been tried to be explained.

Key words*: raw materials for cellulose; industrial forest; plantation forest; Forest Property in Turkey; pulp product in Turkey.*

INTRODUCTION

Forests, which have a vital importance for all living creatures, have a great and indispensable place in industry as well as the beauty and oxygen they provide. The trees that make up the forests are very diverse in terms of species. This is important for wood production, ecosystem and obtaining a wide vaiety of wood raw materials.

Forests are considered as normal forests and degraded forests. Considering that the primary management objective of the forests is the production of wood raw materials, forests where the trees cover less than 10% of the land are considered as "degraded" forests. Closure is an important factor affecting the production of wood raw materials of forests. Closure is an important variable that affects the production of wood raw materials of forests. For this reason, such forests are referred to as degraded in terms of wood production and are expected to be transformed into normal forests (10th Development Plan 2014-2018). There are more than 10 thousand plant species in Turkey. Three thousand of them are endemic. There are also 150 species of trees called forest trees (OGM 2006). These 150 varieties (conifers) are superior to the others in terms of area and increment.

Industrial wood production, which had been stable for a long time, has gained momentum in recent years. Firewood production has reduced.

On the other hand, timber production is evaluated under the title "forest products" in the following publication titled "Forest Products 2011-2012 Market Evaluation" published by FAO and UNECE TC in October, 2012.

Wood as raw material (Timber, industrial wood, firewood etc.) 2. Chipping-board sector 3. Paper, cardboard and wood pulp sector 4. Wood energy sector 5. Carbon sector 6. Sectors with a high added-value (furniture, wooden houses, lamina coated timber) 7. Innovative products (bioplastic, biologic-based products, biorefinery-biogas.

The effectiveness of individuals and institutions that use wood or other forestry products as their main raw materials and increase the degree of utility by changing the shape of these products is closely related to both the country's economy and the sustainability of forests.

The legislation on private forestry in Turkey first started with the law number 3116 dated 1937. Although incentives have been granted in terms of of private forestry, the rate of private afforestation has remained low compared to the public sector. The economic and social conditions of the country affected this. Especially in cities where the alternative cost of land has increased, some forest owners have tried to transform their private forests into settlements while private entrepreneurs who have reached a certain level of prosperity have made examples of social and economic forestations.

Forest and forest industry are closely related sectors. The wood raw material produced by the forest has always been an important source of financing for the sector as well as increasing the importance of other goods and services. Although it is seen that private business is dominant in the forest industry; it is thought that there is no good coordination between the forest and the industry.

In recent years, due to the closure of cellulose producing factories in Turkey, paper production is based on imports.

OBJECTİVES

The main objective of the present research was the importance of industrial forests in cellulose production and the current situation in Turkey has been tried to be explained.

METHOD, MATERİALS AND EQUİPMENT

It is revealed that our country's wood raw material demand, which was 30 million m^3/year, will increase to 61m^3/year in 2020, and there will be 40 million m^3 deficit between production and consumption. It is pointed out that the best way is to focus on industrial plantations as replacement goods and import will not solve the problem completely because this deficit will be impossible to meet by our natural forests (Birler 1995). The idea of establishing an industrial plantation came to the forefront first at the beginning of 1950s and the first step was taken by F. Fırat with the seeds of Duglas brought from France in 1951 (Asan 1989). This idea was gained a corporate identity with the "Poplar and Fast Growing Forest Trees Research Institute" established in Izmit in 1962 (Boydak 1998). Starting from 1968, this institute has been established in 40 provinces in coastal areas from Rize to Kahramanmaras, with extensive domestic and foreign rapid developing species.

With the project "Tur 71/521 Industrial Forestry Plantations", which was put into practice in 1972 and completed in 1978, this idea, which has gained widespread popularity in Turkey, has reached an important stage with massive afforestation in the second half of 1970s. A total of 39 permanent test sites have been set up in the Aegean (9), Marmara (19), Black Sea (4) and Mediterranean (7) regions during these years (Asan 1998; Öztürk 1995).

These afforestation works reached 100.000 ha/year in the second half of 80's but declined due to various financial incapabilities in 90's. (Ürgenç et al.1993).

One of the important developments that took place in 2004 was the establishment of a company named "Ağaç Tarımı Anonim Sirketi" by TEMA Foundation. The aim of this company is to "make fast growing tree species and industrial forest plantation, and to do the necessary cultivation and research of seeds and saplings, to establish nurseries, to sell firewood and to make wood trade".

According to a comparison with natural forests in countries where industrial plantations play an important role and this kind of forestry is practised, wood production in these countries is as follows; 22m^3/ha/year in New Zealand, 20m^3/ha/year in Chile and Indonesia. This shows how important plantation forestry is in order to meet wood deficit.

Up to now, plantation studies in our country have produced 125 000 hectares of poplar plantations, including 65 000 hectares of hybrid poplars (Populus x euramericana) and Populus deltodies and 60 000 hectares of black poplar.

Eucalyptus afforestation (Eucalyptus camadulensis and E.grandis) is 20 000 hectares. Industrial forest afforestation with leafy species is 145 000 hectares (except alder, ash and similar leafy afforestation). The industrial afforestation with fast-growing seeds is about 55 700 hectares (Çalışkan 1998). Of this afforastation, 53 901 ha is maritime pine (Pinus pinaster). Other species are Pinus radiata (1642ha), Pseudotsuga menziessii (140ha) and Pinus taeda (17ha). The sum of the industrial forest afforestation which is made up to date in Turkey is about 205 000 hectares. Most of these plantations are of the classical afforestation types established by species that are growing fast rather than industrial plantations (Boydak and Çalışkan 2014).

Black poplar (Populus nigra), Fırat Poplar(Populus euphratica), populus tremula, willow species (Salix sp), ash tree (Fraxinus excelsior), alder (Alnus glutinosa, A. Glutinosa subsp, Barbata), plane tree (Platanus orientalis), chestnut (Castane sativa), Calabrian pine (Pinus brutia), black pine (Pinus nigra subsp.Pallasiana) and Abies equi-trojani are natural and fast-growing species of our country.

Fast-growing foreign species which were tested and whose plantations were established in Turkey are hybrid poplars (Populus x euramericana), P.deltoides, Eucalyptus camaldulenis, Eucalyptus grandis, Pinus pinaster, Pinus radiata, Pinus taeda and Pseudotsuga menziesii (Boydak and Çalışkan 2014).

The tree existence, the area covered by trees, and the increment values of tree species suitable for the production of cellulose such as calabrian pine, black pine, yellow pine, fir, ladin, maritime pine, beech, poplar, eucalyptus and locust are given in Table 3.1. As the table shows, the highest increment

is obtained from black pine with 9 830 865m³ and broad-leaved beech tree with 9 298 396 m³. This is followed by calabrian pine and poplar, with a little more than half of these increments.

Table1

Areas, existence and increments of our tree species which are suitable for cellulose production

Types of Trees	Area (ha)	Tree Riche (m^3)	increase (m^3)
Pinus brutia Ten(Pinaceae)	5 854 672,8	286 242 133	8 820 737
Pinus nigra Arnold.(Pineceae)	4 693 059,6	342 887 462	9 830 865
Pinus sylvestris L. (Pineceae)	1 479 647,6	135 724 263	3 304 266
Abies cp. (Pineceae)	670 389,6	117 991 686	2 715 872
Picea (Pineceae)	334 472,4	62 254 789	1 453 060
Pinus pinaster	63 668,1	5 697 829,8	345 000
Fagus sp. L. (Fagaceae)	1 961 659,5	343 661 017,4	8 298 396
Populus spp. (Salicaceae)	6 546,5	140 356	16 281
Eucalyptus	2 528,2	28 496	2 099
Robinia	64,6	4 484	180
Total	**15 066 708,9**	**1 294 632 516**	**34 786 756**

Source: OGM, General Directorate of Forestry, Department of Forest Management Planning, 2012 Inventory Results

Plantatıon Forests

The increasing need for wood can not be met by our existing natural forest resources, and the raw and imported wood raw materials are not sustainable and cheap. Work has been done to establish plantation forests for this. This work; T. C. A project has been initiated in the technical assistance and organization of the Ministry of Environment and Forestry, the TEMA Foundation, Istanbul and Düzce Forestry Faculty's contributions, İzmit-Poplar and Fast-Growing Forest Trees Research Institute. This project was initiated on 28.03.2005 by the establishment of ENAT A.Ş (Industrial Tree Farming Industry and Trade Incorporation Company).

The aim of the project "Establishment and development of industrial forest plantations in Turkey" is to support the supply of raw materials by reducing the foreign source dependency of the industrial enterprises that use wood as raw material. In this project named as ENAT A.Ş, the number of partners increased from 26 to 29 in 6 years. Among the partners are companies such as Kastamonu Entegre A.Ş, Çamsan A.Ş, Oyka Kağıt Ambalaj Sanayi ve Ticaret A.Ş and Yonsan Ege Yonga Levha Sanayi ve Ticaret A.Ş.

Afforestation activities officially started in May, 2005 with the land procurement studies. The first afforestation work was started in Karacabey - Kiranlar village. Firt seedling in a total of 1480 acres of afforestation area were planted on the ground in the year 2005, after the cover cleaning and soil treatment with dozer tractor. Approximately 100 people worked each day for a total of three months and 313 thousand pine tree planting and maintenance work was completed. In order to obtain the highest yield per unit area and produce thin wood which fibre-chip and paper industry needs, half of the saplings will be cut 8 or 9 years after the first planting so that both midproduct will be obtained and remaning units will grow. After 16 or 18 years, the final products will be obtained and the field will be afforested again. The aim of the company is to perform industrial afforestation in 300-hectare field.

Plantation forestry is important both for forest existence and sustainability. While natural forests can provide a wood increment of 1-2m^3/year on 1 hectare area per year, the same plantation forest provides at least an increment of 102m^3/year. In addition, the duration of administration in natural forests is between 80 and 100 years, while in plantation forests this time falls to 10-30 years.

In afforestation of plantation forests, species are selected to get the best yield in the shortest time possible. Working with fast-growing coniferous trees started in 1950's in Turkey. A comprehensive tree breeding project was carried out in cooperation with the Ministry of Forestry and the United Nations Food and Agriculture Organization (FAO) with the project "Industrial Forestry Afforestation" between 1972 and 1977. In these studies, many origin tests have been conducted in Black Sea, Marmara, Aegean and Mediterranean regions.

Test areas and origins established in coastal regions of Turkey between 1972 and 1977 are presented in Table 2. The highest origin number belongs to pinus pinestar, pinus brutia and pinusradiata. Accordingly, the number of tests on these species is higher compared to other species.

Table 2

Species and their origins which took place in tests between 1972-1977 in coastal regions of Turkey

Species	Number of origin	Number of try	Species	Number of origin	Number of try
Pinuspinestar	17	39	*Pinushalepensis*	3	5
pinusradiata	9	29	*Pinus silvestris*	4	10
Pinusmuricata	4	5	*Pinusnigra. Corsica*	1	1
Pinusconcorta	3	4	*Pinusnigra. Calabrica*	1	2
Pinusponderosa	5	9	*Cupressusarizonica*	4	10
Pinusdensiflora	1	2	*Cupressussempervirens*	2	2
Pinustaeada	4	5	Lübnan Sediri(*Cedruslibani)*	5	9
Pinuselliottii	2	9	*Cedrusdeodara*	3	6
Pinuscaribaea	1	3	*Cedrusatlantica*	2	3
Pinusjeffreyi	2	1	*Pseudotsugamenziessii*	8	9
Pinusvirginiana	2	1	*Abiesbornmülleriana*	2	9
Pinusechinata	1	2	*Larixeurolepis*	1	1
Pinuscanariensis	1	3	*Larixleptolepis*	1	1
Pinuselderica	2	7	*Sequoiasempervirens*	1	3
Pinusbrutia	14	22	*Juniperusvirginiana*	1	3
Pinusnigra	8	15			

(Tunçtaner 2007)

Breeding studies conducted in the following years depending on the experimental sites and breeding studies between 1972-1977 were based on Black Sea, Marmara and Aegean Region.

Table 3

Breeding studies by regions

Region	Test area	Species	Age	Volume(m^3/ha)	Increase(m^3/ha/year)
Black Sea	Ünye Asarkaya	*Pinusradiata*	21	366,4	17,4
		Pinuspinaster	21	296,5	14,1
		Pinusnigra	21	125,3	6,0
	Bafra Sarıgüzel	*Pinusradiata*	21	240,1	11,4
		Pinuspinaster	21	218,6	10,4
		Pinusnigra	21	77,6	3,7
	Sinop Bektaşağa	*Pinusradiata*	21	487,5	23,2
		Pinuspinaster	21	517,3	24,6
		Pinusnigra	21	121,3	5,8
Marmara	Kandıra Kefken	*Pinusradiata*	20	230,5	11,5
		Pinuspinaster	20	223,8	11,2
		Pinusnigra	20	104,2	5,2
	İzmit Işıktepe	*Pinusradiata*	21	160,2	7,6
		Pinuspinaster	21	132,2	6,3
		Pinusnigra	21	52,8	2,5
	Demirköy İğneada	*Pinuspinaster*	21	431,0	20,5
		Pinusnigra	21	172,2	8,2
	Vize Sergen	*Pinuspinaster*	21	208,6	9,9
		Pinusnigra	21	184,3	8,8
Aegean	Söke Ninemsuyu	*Pinusbrutia*	23	142,2	6,2
		Pinuspinaster	23	92,2	4,0
		Pinuspinea	23	107,6	4,7
		Cupressusarizonica	22	19,8	0,9
	Kuşadası Küçükakdere	*Pinusbrutia*	23	235,5	10,2
		Pinuspinaster	23	294,4	12,8
		Pinuspinea	23	189,1	8,2
		Cupressusarizonica	23	351,5	15,2
	Yatağan Yumaklı	*Pinusbrutia*	23	85,8	3,7
		Pinuspinaster	23	144,3	6,3
		Pinuspinea	23	90,2	3,9
		Cupressusarizonica	23	63,5	2,7

Source: OGM, General Directorate of Forestry, Department of Forest Management Planning, 2012 Inventory Results

When all these research findings are evaluated, the species that should be used according to the regions are as follows.

In the Aegean and Mediterranean regions, Pinus brutia is the most suitable one but the Eucalyptus camaldulensis could also be successful in appropriate fields.

Pinuspinestar is the most successful species in Marmara Region. Additionally, if the suitable growing environment is provided, pinusradiata, pinusbrutia and alnus barbata planting can also be performed.

Pinuspinestar is the most successful species in Western and Central Black Sea regions. In addition to maritime pine, pseudotsugamenzessii can also be planted successfully. Also, in appropriate growing conditions, pinusradiata and alnusglutinosasubsp. Barbata) are also recommended.

Existing plantation forests are summarized in Table 4.

Table 4

Plantation areas and regions of pine and eucalyptus species in Turkey.

Türler	Bölge	Alan(ha)
Pinustaeda	Kerpe-Marmara	17
Pinusradiate	Kerpe-Marmara	1692
Pseudotsugamenziesi	Sinop-Karadeniz	140
Pinuspinaster	Kerpe-Marmara	53901
Okaliptuscamaldulenis	Tarsus-Akdeniz	3263

Akkayan et al. 1983

CONCLUSION

For the sustainability of plate production and cellulose production based on forest products, forest and forest industry should act together. The reason why there is no improvement in the utilization of the forest presence in terms of cellulose production is because cellulose production continues with a single factory. Since the cellulose factories are closed, the necessary cellulose is supplied by import. For this, cellulose production is available from countries such as USA, Canada, China, Brazil, Sweden, Finland, Russia, Portugal.

The fact that the production of cellulose in Turkey is very low and there has been improvements in the production of plate products, which are known as industrial transformations, has produced a result which compensates each other. As in the case of cellulose production, any production-related problem in plate production will cause to a difficulty in finding market for thin wood raw material. For this reason, it is important to examine future changes in terms of both forest management and forest industry as well as macroeconomic impacts. As a result, it would be beneficial for those who manage the forests and those who manage the forest industry to work in coordination, considering international competition. The contribution of plantation forestry to the sustainability of the forests' presence is also significant, as is the yield and quality of cellulose. The leading countries in cellulose production also give importance to plantation forestry. For this reason, these countries usually cultivate eucalypt and populus species, which are important for cellulose production, from leafy trees in private forests and use them in the production of cellulose.

Establishing industrial forests for the development of paper and packaging sector and to facilitate their access to raw material will increase Turkey's forestry presence. In our country, a poplar project work was conducted mainly for the production of cellulose, but with the government policies and the closure of the cellulose factories, the project was not carried into effect. In this project; a study was carried out on the biogenetically developed poplar populations by TUBITAK, Izmit Poplar Research Institute, Istanbul University Faculty of Forestry, Istanbul University Faculty of Science and SEKA (Turkey Cellulose and Paper). The aim was to develop poplar whose lignin is low, cellulose yield is high and duration of management (cutting time) is short. The next step of the project was to encourage the growth of poplars in private forests with government procurement guarantees. Although the results of the project were positive, the cellulose factories were closed, so this project were not put into practice. Today, China is importing paper from Turkey since a production facilitity in China was closed even without evacuating the machines. The production, which was 40 million tons, showed a decrease of 20 thousand tons. Although it is not a big number for China, this means a new market for Turkey, which should be considered.

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THE EVOLUTION OF CHILDREN'S FURNITURE

Emine Seda ERDINLER
Istanbul University, Faculty of Forestry
34473 Istanbul, Turkey
Tel: 0090 212 3382400, Fax: 0090 212 2261113, E-mail: seda@istanbul.edu.tr

Derya SEVİM KORKUT
Duzce University, Faculty of Forestry
81620, Duzce, Turkey
E-mail: deryasevimkorkut@duzce.edu.tr

Kucuk Huseyin KOC
Istanbul University, Faculty of Forestry
34473 Istanbul, Turkey
E-mail: hkoc@ istanbul.edu.tr

Abstract

The paper presents the evolution of children's furniture within the history. Reaching to the present times the children's furniture now has various considerations. Kid's furniture was considered as a place to sleep and a place to keep the baby safe in place. In the older days the children used to sleep with their parents. The first furniture for babies and children was manufactured in the 19th century. Cribs and basinets were the preliminary furniture for infants to sleep. Over time more types of furniture were created for the safety issues. Changing table, high chair, baby walker, toy chest were the examples for the furniture pieces. In the present modern world, safety of a child is important, on the other hand psychology is very important, too. The bedroom of a child is the place where the child spends most of his/her time at home. The environment where the child grows affects the future behaviours and attitudes. The rooms of the children determine the things of amusement and instruction. In this study information from the previous studies were used to determine the past and present situation of children's furniture. Surveys and interviews with the manufacturers are planned for the next stage of this study.

Key words: *furniture; children's furniture; furniture and child development; ergonomics; safety.*

INTRODUCTION

Furniture styles have changed in cycles. The design and other attributes have been affected by decorative trends. The children's furniture have also changed during time. The children used to sleep with their parent until the 19th century. For safety of the children the need for a separate furniture occurred. The modernization in the society also brought awareness to other issues for the children. Furniture became important both for safety and for the development of the children. Soyupak (2015) mentioned the importance of furniture as the children interact with his/her surrounding artificial environment via the furniture as the furniture pieces are the elements forming the environment in the children's room.

In the study by Buyukpamukcu (2004), it is mentioned that as the children grow up, their needs and expectations change. Therefore, it is important to consider their preferences when designing their environment. Environments for children, to live, play and learn is the place for them to make their own decisions.

Sofuoglu et al. (2014) stated 3 basic factors to consider while choosing the furniture for children; the anthropometry of the children, design properties of the furniture such as round corners, functionality, colour, etc., and the effects of the furniture material on health.

According to the study by Demirarslan and Aytore (2004) colourful and active rooms constitute the first step for raising a talented and freethinking.

Physical sizes for different ages, safety for children, children's cognitive ability, furniture decoration, environmental design make up the Human Factor of children's furniture according to the study conducted by Dai and Xu (2013). In the same study, as home being the main place in which children develop and form their unique character, the social segmentation of furniture design occurred as a need.

Faizi et al. (2012), mentioned the importance of natural and artificial environment for increasing the creativity of the children.

In the study of Bulbul et al. (2014) focused on the dangers of furniture, the researchers pointed out the safety considerations and importance of ergonomics.

The manufacturers of children's furniture should consider both ergonomics and social needs of the children while designing their products. The families play an important role on choosing the furniture for their children. Therefore the families should also consider the needs of their children. In the study of Hasturk (2012) parents make the final decision searching for the best price, safety and ergonomics where psychologic approach stands on the back.

From the view of the manufacturers there are more issues to consider. Ecological and multifunctional designs make the present considerations for the manufacturers.

OBJECTIVE

The main objective of this study is to assimilate ergonomic safety and psychological effects in the children's room. Safety as the primary important fact in children's furniture production should be accompanied with the facts affecting a child's development. A further study is planned to determine the approach of the enterprises and designers of children's furniture.

MATERIAL AND METHOD

In this work, approaches for children's furniture were studied referring to the earlier studies. There are various points of view about children's furniture design and production. Some researchers focus on the safety and ergonomic issues, on the other hand some researchers focus on the psychological effects of the bedroom of a child where furniture make up the artificial environment. Creating the proper environment for the child requires proper selection of furniture and effective organization of the furniture.

DISCUSSION

In the early centuries the children did not have a separate space. The spaces were for adults and the children were a part of the place. The sleeping and playing area of the children were the same place as the parents' sleeping place and living environment. However, sleeping with parents was not safe for the children, especially for the infants. Therefore, the need for a separate furniture for children occurred. The cribs and bassinets were the primary furniture pieces designed for children.

Pieces such as changing table, high chairs, night stands, dressers, chests were added in time as the children started to have a separate space in the house.

Safety has been the first consideration before design when it is about the children. Standards for children's furniture have been established for the manufacturers. The regulations about the distance between side uprights and the regulations about the finishes are the most mentioned standards for the safety of the children. The distance between the side uprights must be built close enough so that the child's head would not go through (TS EN 1130-1/2). Non-toxic and lead free finishes must be used as teething babies would chew the edges (Furniture Facts 2005).

Sides and corners must be rounded and there must be no sharp edges (Saka 2017).

There are helpful tips for the consumers of children's furniture in the present studies. These recommendations are more about the safety. Below is the list for the consumers to consider while buying the furniture for their child (Bulbul et al. 2014).

- Certain standard label and documents about raw materials, ergonomics and safety must be available.
- Furniture construction must be durable.
- Non oxidant paint must be used for the metal parts.
- Plastic parts must be attached firmly.
- Wheels must have safety locks.
- The corners must be smooth, no sharp edges.

Although there are various studies emphasizing the importance of safety, there are studies about the importance of the psychological effects of children's furniture.

The children's bedroom is the place where they grow and spend most of their childhood period. This period is very important to gain their future behaviours and attitudes. Children actively participate in their own development. Motor coordination in young children develops along with muscular strength and speed. Through active play, young children learn to channel strength and the speed into smooth, accurate movements. These developments take place in the physical environment of the child. The basic environment of the child is the bedroom. The children's furniture make up the physical environment for the child.

Sleeping is the main activity in the bedroom and it play an important role on healthy growth of a child. The rooms designed for children constitute their environment. The bedrooms of children determine the facts of amusement and instruction for them. Physical stimulation for cognitive and social development is provided by furniture, toys, books and physical objects (Buyukpamukcu 2004).

Effects of colour on personal behaviour is well-known. In children's furniture, effect colour plays an important role. Warm colours such as red, yellow and orange activate a child. The cool colours such as blue and green make a calm down effect on the children. Heseltime and Halborn as sited by Buyukpamukcu (2004) stated that the colours incorporated with the furniture design affects the stimulation of reinforcement of the feelings of the children in their bedroom.

According to the study by Faizi et al. (2012), colours had a significant correlation with creativity potential. Pleasant, colourful and exciting images and items have been considered as the basis for motivation of creativity.

CONCLUSIONS

In this work, the evolution of children's furniture was studied. The recent studies show that the children's furniture pieces make up the environment of the child. The environmental factors are important for the development of the child.

Beginning from the 19th century, the approach of furniture for children has changed. It is not only the safety regulations to be considered, psychological needs of the children for their development of attitudes and behaviours must be taken into consideration while designing and manufacturing children's furniture.

It is important to raise awareness on the manufacturers and parents to consider both safety and developmental factors.

Professional view of a psychologist would bring different perspectives in the design decisions. Therefore it would be helpful to employ a psychologist or supply a psychologist advisor in the enterprises manufacturing children's furniture.

The parents can also get design ideas from psychologists to form their choice of bedroom for their children.

Further stage of this study is planned as the field study. The opinions and approaches of Turkish furniture manufacturers to the children's furniture design, production and marketing will be in the research concept.

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LIST OF PARTICIPANTS

NAME	TITLE	INSTITUTION	E-MAIL ADDRESS
Akca Mehmet	Dr.	Balikesir University, Dursunbey Vocational School, Balikesir, Turkey	makca@balikesir.edu.tr
Alfredsen Gry	Prof.dr.	Norwegian Institute of Bioeconomy Research, Norway	gry.alfredsen@nibio.no
Angelova Desislava	Assoc.Prof.dr.	University of Forestry, Sofia, Bulgaria	d_i_angelova@abv.bg
Angelski Dimitar	Assoc.Prof.dr.	University of Forestry, Sofia, Bulgaria	d.angelski@gmail.com
Antal Reka Maria	Dr.	Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts, Sopron, Hungary	reka.maria.antal@skk.nyme.hu
Antonoaie Cristina	Lecturer dr	DRESMARA, Romania	cristina.antonoaie@gmail.com
Atanasov Valentin	Assoc.Prof.dr.	University of Forestry - Sofia, Faculty of Forest Industry, Department of Woodworking Machines, Bulgaria	vatanasov_2000@yahoo.com
Atar Ilkay	PhD student	Kahramanmaras Sutcu Imam University, Turkey	iatar@ksu.edu.tr
Ay Nurgul	Prof.dr.	Karadeniz Technical University, Trabzon, Turkey	nay@ktu.edu.tr
Bader Matyas	PhD student	Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts, Sopron, Hungary	bader.matyas@phd.uni-sopron.hu
Badin Nicolae	Eng. PhD student	KASTAMONU Reghin, Romania Transilvania University of Brasov, Faculty of Wood Engineering, Romania	nicolae.badin@kastamonu.ro

Bak Miklos	Dr.	University of Sopron, Simonyi Karoly Faculty of Engineering, Hungary	bak.miklos@uni-sopron.hu
Barboutis Ioannis	Prof.dr.	Aristotle University of Thessaloniki, Greece	jbarb@for.auth.gr
Barbu Marius Catalin	Prof.dr	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	marcat@gmx.at; cmbarbu@unitbv.ro
Barbuta Costel	Dr.	FPInnovations, Quebec, Canada	costel.barbuta@fpinnovations.ca
Barmpoutis Panagiotis	Dr..	Aristotle University of Thessaloniki,Faculty of Agriculture, Forestry and Natural Environment, Greece	panbar@auth.gr
Bartha Biborka	PhD student	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	bartha.biborka@unitbv.ro
Bayatkashkoli Ali	Dr.	University of Zabol, Iran	ali.bayatkashkoli@gmail.com
Bedelean Bogdan	Lecturer dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	bedelean@unitbv.ro
Bekhta Pavlo	Dr.	Ukrainian National Forestry University, Ukraine	bekhta@nltu.edu.ua
Beldean Emanuela	Lecturer dr	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	ebeldean@unitbv.ro
Birinci Emre	PhD student	Kastamonu University, Turkey	ebirinci@kastamonu.edu.tr
Blomqvist Lars	Dr.	Linnaeus University,Department of Building Technology, Sweden	lars.blomqvist@lnu.se
Brenci Luminita	Assoc.Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	brenlu@unitbv.ro
Buchner Julia	PhD student	Ecole Superieure du Bois, Nantes, France	julia.buchner@ecoledubois.fr

Budau Gavril	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	g.budau@unitbv.ro
Campean Mihaela	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	campean@unitbv.ro
Candrea Adina Nicoleta	Lecturer.dr.	Transilvania University of Brasov,Faculty of Economic Sciences and Business Administration Romania	adina.candrea@unitbv.ro
Cionca Marina	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	marinacionca@unitbv.ro
Cismaru Ivan	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	icismaru@unitbv.ro
Comsa Ghiorghita	Lecturer dr	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	comgn@unitbv.ro
Coole Tim	Dr.	Bucks New University, UK	Tim.Coole@bucks.ac.uk
Cosereanu Camelia	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	cboieriu@unitbv.ro
Couceiro Jose Miguel	PhD student	Lulea University of Technology, Skellefteå, Sweden	jose.couceiro@ltu.se
Csiha Csilla	Dr.	University of West Hungary, Sopron, Hungary	csiha.csilla@uni-sopron.hu
Del Menezzi Cláudio	Assoc.Prof.dr.	University of Brasília, Faculty of Technology, Brazil	cmenezzi@unb.br
Denes Levente	Dr.	Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts, Sopron, Hungary	levente.denes@skk.nyme.hu
Diaz-Maroto Ignacio J.	Dr.	Universidad de Santiago de Compostela, Spain	ignacio.diazmaroto@usc.es

Dogan Kadir	PhD student	SFC Integrated Wood Products, Kastamonu, Turkey	kadir.dogan7137@gmail.com
Erdinler Emine Seda	Assoc.Prof.dr.	Istanbul University, Turkey	seda@istanbul.edu.tr
Filippou Vasileios	PhD student	Aristotel University of Thessaloniki, Greece	vafilpo@gmail.com
Fodor Fanni	PhD student	Institute of Wood Science, Simonyi Karoly Faculty of Engineering, Wood Sciences and Applied Arts, Sopron University, Hungary	fodor.fanni@phd.uni-sopron.hu
Fotin Adriana	Lecturer dr	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	adrianafotin@unitbv.ro
Franke Tom	Researcher	Eberswalde University for Sustainable Development, Eberswalde, Germany	tom.franke@hnee.de
Fredriksson Magnus	Dr.	Lulea University of Technology, Sweden	magnus.1.fredriksson@ltu.se
Frömel-Frybort Stephen	Dr.	Wood K plus Linz, Austria	s.frybort@kplus-wood.at
Gaff Milan	Assoc.Prof.Dr.	Czech University of Life Sciences, Prague, Czech Republic	gaffmilan@gmail.com
Geng Jing	MSc	Beijing Forestry University, China	eliza2012@bjfu.edu.cn
Georgescu Sergiu	PhD student	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	sergiu.georgescu@unitbv.ro
Gezer Engin Derya	Assoc.Prof.dr.	Karadeniz Technical University, Turkey	engin_gezer@yahoo.com
Gochev Zhivko	Assoc.Prof.dr.	University of Forestry, Sofia, Bulgaria	zhivkog@yahoo.com
Goncalves Carolina	PhD student	University of Porto, Faculty of Engineering, Porto, Portugal	carolina.goncalves@sonaearauco.com

Grecu Virgil	Prof	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	vgrecu@unitbv.ro
Grepcka Alketa	Dr.	Agricultural University of Tirana, Albania	agrepcka@ubt.edu.al
Gurau Lidia	Assoc.Prof	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	lidiagurau@unitbv.ro
Hacibektasoglu Murat	Eng. PhD student	Yildiz Entegre Romania Transilvania University of Brasov, Faculty of Wood Engineering, Romania	murathacib@hotmail.com
Hansson Lars	Assoc.Prof. dr.	Norwegian University of Science and Technology (NTNU) Alesund, Norway	laha@ntnu.no
Hiziroglu Salim	Prof.dr.	Oklahoma State University, USA	salim.hiziroglu@okstate.edu
Hodgson Stephen	Chief executive officer	Property Care Association, UK	steve@property-care.org
Horacek Peter	Prof.dr.	Czech University of Life Sciences, Prague, Czech Republic	petrtomashoracek@seznam.cz
Ionescu Constantin Stefan	Expert restorer, PhD student	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	restaurareionescu@gmail.com
Irbe Ilze	Dr.	Latvian State Institute of Wood Chemistry, Latvia	ilzeirbe@edi.lv
Irle Mark	Dr.	Ecole Superieure du Bois, Nantes, France	mark.irle@ecoledubois.fr
Ishii Sachio	General manager	EDS Laboratory, Japan	info@eds-lab.jp
Ispas Mihai	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	ispas.m@unitbv.ro
Kalaycioglu Hulya	Prof.dr.	Karadeniz Technical University, Turkey	khulya@ktu.edu.tr

Kamperidou Vasiliki	Dr.	Aristotle University of Thessaloniki, Greece	vkamperi@for.auth.gr
Karakus Kadir	Assist.Prof.dr.	Kahramanmaras Sutcu Imam University, Turkey	karakus@ksu.edu.tr
Kaymakci Alperen	Assist.Prof. dr.	Kastamonu University, Turkey	akaymakci@kastamonu.edu.tr
Koutsianitis Dimitrios	Dr.	Aristotle University of Thessaloniki, School of Forestry and Natural Environment, Greece	koutsian@for.auth.gr
Kraler Anton	Assoc.Prof.dr.	University of Innsbruck, Timber Engineering Unit, Innsbruck, Austria	anton.kraler@uibk.ac.at
Lengyel Karoly	Eng. PhD student	KASTAMONU Reghin, Romania Transilvania University of Brasov, Faculty of Wood Engineering, Romania	Karoly.Lengyel@kastamonu.ro
Lenz Claudia	MSc	Eberswalde University for Sustainable Development, Eberswalde, Germany	claudia.lenz@hnee.de
Lindblad Fredrik	Researcher	Linnæus University, Sweden	fredrik.lindblad@lnu.se
Lunguleasa Aurel	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	lunga@unitbv.ro
Macchioni Nicola	Researcher	CNR - IVALSA, Italy	macchioni@ivalsa.cnr.it
Mengeloglu Fatih	Prof.dr.	Kahramanmaras Sutcuimam University, Turkey	fmengelo@ksu.edu
Mihajlova Julia	Assoc.Prof.dr.	University of Forestry, Faculty of Forest Industry, Sofia, Bulgaria	jmihajlova@ltu.bg
Millis Susan	Researcher	Private researcher, UK	smmillis@hotmail.com
Mitani Andromachi	Dr.	Aristotle University of Thessaloniki, Greece	amitani@for.auth.gr
Moldoveanu Iuliu	Eng.	Romanian Furniture Club Association Bucharest, Romania	iuliumoldoveanu@acrm.ro

Morcan Ciprian	Eng.	Transylvania Furniture Cluster Cluj-Napoca, Romania	ciprian.morcan@mobiliertransilvan.ro
Moutou Pitti Rostand	Dr.	Université Clermont Auvergne, CNRS, Institut Pascal, France	Rostand.moutou_pitti@univ-bpclermont.fr
Muscu Ioan	Assoc.Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	ioanmuscu@yahoo.com
Nemeth Robert	Prof.dr.	University of Sopron, Hungary	nemeth.robert@uni-sopron.hu
Olarescu Alin	Lecturer dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	a.olarescu@unitbv.ro
Olaru Valeriu Ion	Expert restorer	CNM ASTRA Museum Sibiu, Romania	valeriuolaru.astra@yahoo.com
Olorunnisola Abel	Prof.dr.	University of Ibadan, Nigeria	abelolorunnisola@yahoo.com
Osvaldova Makovicka Linda	Dr.	Department of Fire Engineering, Faculty of Security Engineering, University of Zilina,Slovakia	linda.makovicka@fbi.uniza.sk
Özbay Gunay	Assoc.Prof.dr.	Karabuk University, Turkey	gozbay@karabuk.edu.tr
Ozdemir Turgay	Prof.dr.	Karadeniz Technical University, Turkey	turgay@ktu.edu.tr
Ozden Oznur	Dr.	Istanbul University, Faculty of Forestry, Istanbul, Turkey	ozdeno@istanbul.edu.tr
Pal Zsuzsanna	Eng.	Imageline Odorheiu Secuiesc, Romania	zspal@imageline.ro
Petric Marko	Prof.dr.	University of Ljubljana, Biotechnical Faculty, Slovenia	marko.petric@bf.uni-lj.si
Petru Adrian	PhD student	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	adrianpetru_ro@yahoo.com
Pfriem Alexander	Prof.dr.	Eberswalde University for sustainable development, Germany	alexander.pfriem@hnee.de

Pop Ion Octavian	Dr.	University of Limoges, France	ion-octavian.pop@unilim.fr
Porojan Mihaela	Lecturer dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	mporojan@unitbv.ro
Racasan Sergiu	Assist.dr	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	sergiu.racasan@gmail.com
Rademacher Peter	Prof.dr.	University of West Hungary, Sopron, Hungary	Peter.Rademacher@uni-sopron.hu
Radulescu Loredana	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	loredana21ro@yahoo.com
Raycheva Regina	Assoc.Prof.dr.	University of Forestry, Sofia, Bulgaria	rexi@abv.bg
Reh Roman	Assoc.Prof.dr.	Technical University in Zvolen, Slovakia	roman.reh@tuzvo.sk
Rosca Calin	Prof.dr.	Transilvania University of Brasov, Faculty of Mechanical Engineering, Romania	icrosca@unitbv.ro
Rosu Dan	Dr.	Petru Poni Institute of Macromolecular Chemistry, Iasi, Romania	drosu@icmpp.ro
Rosu Liliana	Dr.	Petru Poni Institute of Macromolecular Chemistry, Iasi, Romania	lrosu@icmpp.ro
Salca Emilia	Assoc.Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	emilia.salca@unitbv.ro
Sandberg Dick	Prof.dr.	Lulea University of Technology, Sweden	dick.sandberg@ltu.se
Savov Viktor	Assoc.Prof.dr.	University of Forestry, Faculty of Forest Industry, Sofia, Bulgaria	victor_savov@ltu.bg
Sawyer Gervais	Researcher	International Wood Products Journal, UK	Gervais.Sawyer@blueyonder.co.uk

Schauerte Tobias	Senior lecturer	Linnæus University, Sweden	tobias.schauerte@lnu.se
Schonfelder Ondrej	PhD student	Czech University of Life Sciences, Faculty of Forestry and Wood Sciences, Prague, Czech Republic	schonfelder.ondrej@seznam.cz
Schreiner Linda	PhD student	Fraunhofer Institute for Process Engineering and Packaging IVV and Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany	linda.schreiner@ivv.fraunhofer.de
Shiga Shigehito	Mr.	EDS Laboratory, Japan	shigashigehito96@yahoo.co.jp
Skog Johan	Researcher	RISE Reseach Institutes of Sweden, Division of Bioeconomy, Skellefteå, Sweden	johan.skog@ri.se
Sova Daniela	Assoc.Prof.dr.	Transilvania University of Brasov, Faculty of Mechanical Engineering, Romania	sova.d@unitbv.ro
Spirchez Cosmin	Lecturer dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	cosmin.spirchez@unitbv.ro
Stanciu Mariana Domnica	Lecturer dr.	Transilvania University of Brasov, Faculty of Mechanical Engineering, Romania	mariana.stanciu@unitbv.ro
Teaca Carmen Alice	Dr.	Petru Poni Institute of Macromolecular Chemistry, Iasi, Romania	cateaca@icmpp.ro
Teppand Tonis	Lecturer, PhD student	Estonian University of Life Sciences, Estonia	tonis.teppand@emu.ee
Tesarova Daniela	Dr.	Mendel University, Brno, Czech Republic	tesar@mendelu.cz
Timar Cristina	Prof.dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	cristinatimar@unitbv.ro
Trischler Johann	Dr.	Lulea University of Technology, Skellefteå, Sweden	johann.trischler@lnu.se

Turcas(Diaconu) Minerva	PhD student	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	minerva2013diaconu@gmail.com
Ustaomer Derya	Assoc.Prof.dr.	Karadeniz Technical University, Turkey	uderya@ktu.edu.tr
Van Acker Joris	Prof.dr.	Ghent University, Belgium	Joris.VanAcker@UGent.be
Varodi Anca	Researcher, dr.	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	anca.varodi@unitbv.ro
Vitchev Pavlin	Assoc.Prof.dr.	University of Forestry - Sofia, Faculty of Forest Industry, Department of Woodworking Machines, Bulgaria	p_vitchev@ltu.bg
Wallenhorst Lena	PhD student	University of Applied Sciences and Arts, Laboratory of Laser and Plasma Technologies, Göttingen, Germany	lena.wallenhorst@hawk-hhg.de
Wang Zhenyu	MSc.	Beijiing Forestry University, Beijiing, China	wangzhenyu1992@126.com
Watkinson Sarah	Dr.	University of Oxford, Department of Plant Sciences, UK	sarah.watkinson@plants.ox.ac.uk
Wimmer Rupert	Prof.dr.	University of Natural Resources and Life Sciences, Tulln, Austria	rupert.wimmer@boku.ac.at
Yang Tiantian	PhD student	Beijiing Forestry University, Beijiing, China	bjfu100514327@163.com
Young Timothy	Prof.dr.	University of Tennessee, Center for Renewable Carbon, USA	tmyoung1@utk.edu
Zeleniuc Octavia	Assoc.Prof. dr	Transilvania University of Brasov, Faculty of Wood Engineering, Romania	zoctavia@yahoo.com