

# 9<sup>TH</sup> Hardwood Proceedings

Part I.

With Special Focus on “An  
Underutilized Resource:  
Hardwood Oriented  
Research”

2020

Sopron, Hungary

Editors:

Róbert Németh,  
Peter Rademacher,  
Christian Hansmann,  
Miklós Bak, Mátyás Báder

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**Constant Serial Editors: Róbert Németh, Miklós Bak**

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## **Influences of some factors upon the accelerated curing of pigmented polyurethane gloss top-coat by UV irradiation**

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**Keywords:** coatings, pigmented finish, veneered MDF, UV dryers

### **ABSTRACT**

Pigmented surface finish is made in a variety of colours and gloss. The most rational method for decreasing the curing time for varnish coatings is through UV irradiation. Many factors however have impact on the process of drying (curing) varnishes. This requires determining the most influential factors to the curing process. The purpose of the current research is to determine the influence of two technological factors over the curing duration of protective decorative coatings via UV irradiation. In this relation, 2-factor experiment was performed so thus to determine the influence of the factors “feeding speed” and “quantity of applying” at UV curing of polyurethane varnish coating over the “curing degree” towards oak veneered MDF. The experiment has been conducted by the usage of equipment for varnish application via spraying followed by drying with two UV lamps (gallium and mercury vapor lamps). The results from those tests were extracted through two scorings that determine the curing degree for the lacquer coating and its adhesion to the veneered MDF.

### **INTRODUCTION**

By curing/drying of wood coatings, most generally it refers to each influence, which causes vapouring of liquids and transit of film coatings from liquid into solid state. The coatings curing is a process, which end is determined via the stage of reached technological hardness by following the quality requirements. At normal operational conditions, i.e. without any external impact, the curing duration of film coatings is for few hours, which is many times longer compared to the operating time needed for completing most mechanical processes, including the operation upon application of materials for film coatings. The modern furniture production achieves sharp reduction of the curing duration for coatings via the usage of lacquer-chemical materials that allow quick transit from liquid into solid state and due to technical equipment and technologies implemented for accelerating the curing process via active forms of external influence. As it is well known, depending on the approach used for curing acceleration, they are divided into 2 main groups: via heating, i.e. entering of heat into applied layer and via radiation-chemical impact into applied layer. The advantages of particular method for curing are estimated in best way when comparing with other methods according to following two indicators: the process continuation from the time of application till reaching the technological hardness; the specific energy consumption for its conduction (Kavalov et al. 2000<sup>b</sup>).

The transit from liquid into solid state usually proceeds gradually. Its externally noticeable performance is the viscosity increase: initially slowly, with short transition from moderate into avalanche fast. Those changes correspond to the transitional states of gelling and glassy state with gradual increase of the hardness of coated film. Simultaneously with the increase of the hardness, there are changes appearing in the sizes of the initially formed liquid layer. Its thickness is decreased; its micro surface is changed as result of the impact of complex relaxation processes (Онегин 1983). Those complex changes play decisive influence on the creation and altering of the time for internal stresses in the coating. They, from their side, affect backward upon the deformation processes, over the initially formed adhesion and cohesion balances in the system coating - surface base. During that second stage, the external view and operational properties of the coating are determined (Kavalov et al. 2000<sup>a</sup>).

The wood coatings have to meet many customer requirements, but the most common are enhance of surface resistance without harmful components and minimizing atmospheric emissions (Yan-jun et al. 2002). UV-curable technology is one of the fastest growing markets in the paint and coating industry. The reasons for the rapid and steady growth of UV-curable coatings are numerous. The most commonly cited are the following: low VOC emissions, excellent mechanical and chemical resistance, and fast curing/drying

(Landry et al. 2015). Upon the usage of a ultraviolet (UV) radiation, its created conditions for multiple (from 20 up to 100 times) shortening of the duration for layers curing, even with thickness exceeding 300  $\mu\text{m}$ . As a result of that, the production area is reduced, the length and price of the curing equipment. Radiation curable coatings need less (if any) additional solvents than conventional coatings, thus being of ecological interest. This coating technology exhibits a number of economic advantages: compact installations, high productivity because of high-speed curing, savings in materials because of excellent film properties of the coatings even in thin layers. Moreover, savings in energy compared to conventional heat curing coatings can be realized, because energy is only needed to initiate curing (Kranig 1993). The methods via radiant-chemical influence reduce multiple times the curing period, but they have much more limited application – mainly for those coatings, which curing is predominantly a chemical process. Only certain types of polymers can be cured by UV radiation because their formulations have to contain unsaturated acrylic and polyester resins (the most common are acrylate-modified polyesters, polyurethanes or epoxy binders) and their copolymers (Bongiovanni et al. 2002, Herrera et al. 2015). Virtually any wood species can be finished to high standards but the selection and use of the proper UV curable coating requires certain considerations (Van Iseghem 2006). The equipment for UV radiation of details with applied coatings are tunnel type. They include certain number of lamps with total or individually organized system for focusing and cooling. The UV lamps used for curing are rated at 120 W/cm per lamp (Tracton 2005). The major parameters that manage the process of photochemical curing are: length of the wave of the top irradiation; the irradiation dose; the irradiation intensity; the distance of the transmitter to the object; thickness and chemical content of the coating. UV sensitive compositions transform into solid state via chemical process initiated by UV rays with wave length from 200 up to 450 nm. When UV varnishes are irradiated with sufficient quantity of UV energy, the photoinitiators in their content get activated and provoke chemical reaction, as a result of which the molecular structures are broken and form free radicals, which in their turn react with the received oligomers and monomers in the substrate and cause fast reaction of spatial networking.

Pigmented surface finish covers the base material and gives a colourful decorative appearance to the product (Slabejová et al. 2018). The curing of pigmented surfaces with UV rays has numerous specifics. It does not flow evenly over the thickness of the finish, thus its hardly applicable for non-transparent coatings. This difficulty could be overcome by using pigments and fillers with increased UV permeability. Despite that, many other factors react over the process of UV curing of coatings (type and quality of liquid compositions, thickness of the liquid film, feeding speed, power, type and location of the UV source, reflectors, emission spectrum of the lamps, etc). The availability of wide range of factors influencing the process of curing of UV lacquered coatings requires to have their influence degree and character being estimated. From practical point of view, the easiest to control is the technological factor ‘feeding speed’. From another side, the protective functions of the coatings depend in high degree on their thickness and respective quantity of applied varnish and paint. The purpose of the current research is to determine the influence of the factors “feeding speed” and “quantity of applying” over the curing duration of polyurethane varnish coating via UV irradiation.

## EXPERIMENTAL METHODS

Fore conducting this research, we have produced sample details from MDF (Medium Density Fibreboard) with dimension size 297x210x20 mm. They have been veneered with oak veneer with 0,6 mm thickness and density of 680 kg/m<sup>2</sup>. After applying 2 layers of white polyurethane primer FPP225/CT7 (produced by Sirca S.p.A. – Italy), the sample details are sanded with sand paper №280. The topcoat of samples is made from white polyurethane glossy varnish - LPP2530NC (produced by Sirca S.p.A. – Italy) with viscosity (DIN 4 at 20<sup>0</sup>C) - 125 s. The coatings are applied on automatic spray coating line with UV curing systems (produced by Venjakob Maschinenbau GmbH & Co – Germany). The line is composed of the following components: dust removal systems; spray booth; drying tunnel with laminar air flow and working temperature- 25<sup>0</sup>C; UV curing tunnel and belt conveyor system. The radiation is generated by two types of UV-curing lamps with different wavelengths and UV-dose: mercury *Hg* (280÷320 nm; 80 W.cm<sup>-1</sup>) lamps to cure transparent and clear UV products and gallium *Ga* (- 390÷450 nm; 120 W.cm<sup>-1</sup>) lamps to cure pigmented UV products.

To determine the influence of the selected technical parameters on the curing duration of polyurethane varnish coating via UV irradiation, 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 and 1999) strongly influencing the curing duration technical factors has been designed and performed. 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). Statistical software “Qstatlab 5” was used for the analysis of the data and calculation of the regression coefficients. In the carried out experiment the values of the variable factors in non-coded form are as follows:

- feeding speed,  $U (X_1) - 1, 4, 7$  m/min;
- Quantity of the applied polyurethane varnish,  $Q (X_2) - 60, 120, 160$  g/m<sup>2</sup>.

In production environment it is necessary to determine the adhesion of the protective-decoration coatings quite fast and with certain punctuality. Usually, for this purpose, the standard method ISO 2409 (2013) is used, which makes cross hatch adhesion test. Coating adhesion of the samples was measured by coating cross-test device (BGD-502/5), according to ISO 2409 (2013). The device is aimed to test coatings with thickness from 60µm until 120µm. The surface of the sample was processed by a multiple-blade cutting tool to form a 6x6 scratch-matrix with 2 mm by 2 mm squares as basic units. The scratches were deep enough to penetrate to the substrate. The cut area was observed with a magnifying glass, and the adhesion of the sample was classified into six grades according to the degree of coating detachment. a six-step classification is given. The first three steps are satisfactory for general purposes (0, 1 and 2) and are to be used when a pass/fail assessment is required (ISO 2409, 2013). The adhesion measurement is done right after application and UV drying of the polyurethane top coating.

The degree of curing change of the obtained coating is reported by determining the reached phase during the curing process. The curing phase or any defect resulting from the curing of the coating are determined organoleptic and visual. The so reported phase is coded into scoring so thus to process further the results from the two-factor experiment. The scoring curing phases for „curing degree" used are as follows: 1 - gelling; 2 - “dust free”; 3 - “touch dry”; 4 - cured and 5-pre-cured (defect in coating adhesion due too high temperature).

## RESULTS AND DISCUSSION

Matrix compositional plan and average values obtained from the experimental test are shown in Table 1. The second order equations from which the regression coefficients have been derived are as follow:

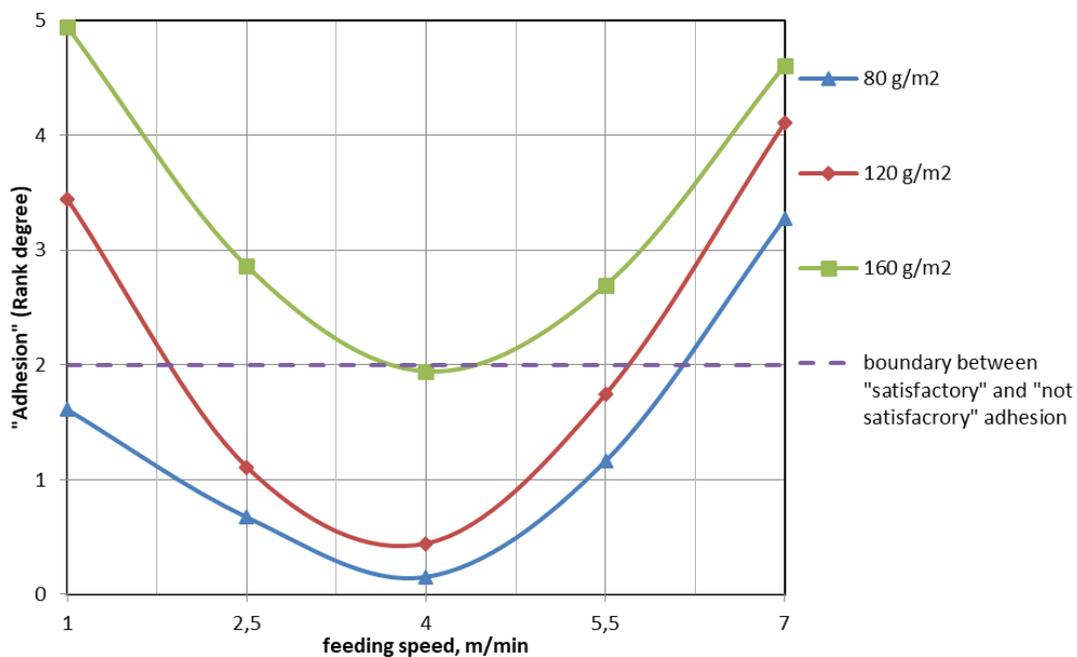
$$Y_{adhesion} = 0,44 + 0,33x_1 + 1,17x_2 + 3,33x_1^2 - 0,17x_2^2 - 0,5x_1x_2 \quad (1)$$

$$Y_{curing\ phase} = 4,11 - 1,33x_1 + 0,17x_2 - 0,67x_1^2 - 0,17x_2^2 - 0,25x_1x_2 \quad (2)$$

**Table 1. Matrix compositional plan and average values from the experimental test**

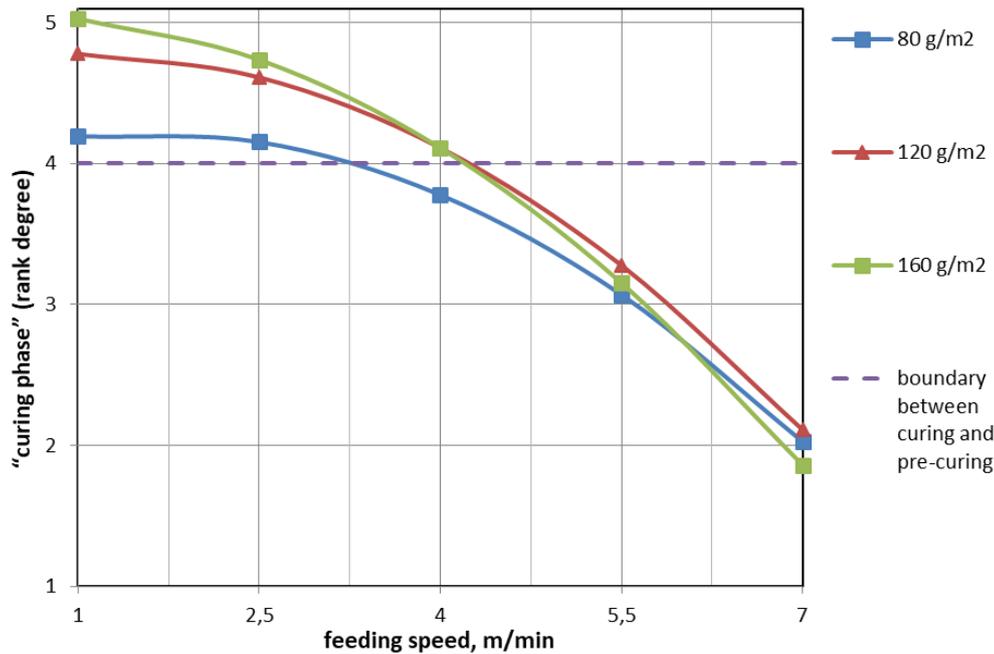
№	U ≡ X <sub>1</sub>	Q ≡ X <sub>2</sub>	Adhesion	Curing phase
1	8 (1)	160 (1)	5	2
2	1 (-1)	160 (1)	5	5
3	8 (1)	80 (-1)	3	2
4	1 (-1)	80 (-1)	1	4
5	8 (1)	120 (0)	4	2
6	1 (-1)	120 (0)	4	5
7	4 (0)	160 (1)	1	4
8	4 (0)	80 (-1)	0	4
9	4 (0)	120 (0)	0	4

From Eq. 1 it is clear that the quantity of applied paint is the strongest factor influencing the adhesion of the coating to the veneered board. Fig. 1 presents the correlation between adhesion and feeding speed of details at UV drying of white polyurethane gloss top-coat varnish. For all used consumption rates (80, 120 and 160 g/m<sup>2</sup>) the coatings adhesion is satisfactory at feeding speed of the details from 3,5 till 4,5 m/min. At feeding speed higher than 5,5 m/min, the irradiation time of the coating is not sufficient for its curing thus comes the weak adhesion of those coatings. The coatings produced at consumption rate of 80 g/m<sup>2</sup> are characterized with very good adhesion. At higher feeding speeds, the curing time of coatings is shortened and thus the process of transformation of the film from liquid into solid state is incomplete (Fig. 2). From the other side, the low feeding speeds lead to the defect called 'pre-curing' of the coating. This defect decreases considerably the coating adhesion especially when using high consumption norm for the varnish application (120 and 160 g/m<sup>2</sup>). For coatings curing at applied consumption rate of over 125 g/m<sup>2</sup>, it is needed to use low feeding speed, but the adhesion of the obtained coatings is rather low. The low feeding speeds result in a defect 'pre-curing' of coatings with thickness over 50µm. This brings to 'unsatisfactory' coating adhesion (Fig. 1). The coatings with thickness up to 50 µm are characterized with good adhesion and their curing is successful even at low feeding speed (1 m/min). The coatings obtained at varnish consumption rate of 160 g/m<sup>2</sup> are characterized with 'unsatisfactory' adhesion. Relatively 'satisfactory' adhesion is achieved just at feeding speed for the details of 4 m/min.



**Figure 1:** Scoring degree of adhesion dependant on the feeding speed for the details. Classification of cross-cut test results: 1 - a cross-cut area not greater than 5 % is affected; 2 - a cross-cut area greater than 5 %, but not greater than 15 %, is affected; 3 - a cross-cut area greater than 15 %, but not greater than 35 %, is affected; 4 - across-cut area greater than 35 %, but not greater than 65 %, is affected; 5 - Any degree of flaking that cannot even be classified by classification 4.

At Fig.2 is presented the correlation between the feeding of the samples and the obtained curing phase for white polyurethane gloss top-coat varnish. To a large extent, the results are similar to the above analysed relations. At feeding speed for the sample details lower than 3,5 m/min is observed the appearance of the 'pre-curing' defect on the coatings. The more is the quantity of applied varnish, the more pronounced is that defect. From the other side, by increasing the feeding speed over 5,5 m/min, the irradiation time of the coatings is not sufficient for their full curing.



*Figure 2: Scoring degree of the UV curing of white polyurethane gloss top-coat varnish dependant on the feeding speed for the details. Curing phases: 1 - gelling; 2 - “dust free”; 3 - “touch dry”; 4 – cured; 5-pre-cured (defect in coating adhesion due too high temperature).*

The obtained results confirm the statement that by coatings with internal pressures, their thickness influences proportionally on the size of the shear stresses, opposing to the adhesion. Respectively, with a considerable increase in the thickness of the coating can be reached internal pressures, which might cause the coating to be peeled from the base. The accumulation of many internal pressures when making thick coatings could be partially prevented by applying increased number of layers, each of them being subject of individual curing (Kavalov et al. 2000<sup>b</sup>). That way, the new layers are formed at partially relaxed basis, thus not accumulating one-way shear stresses on the border line between the coating and its base. The low feeding speeds don't lead to the defect ‘pre-curing’ of the coating at relatively thin coatings (consumption – 80 g/m<sup>2</sup>).

## CONCLUSIONS

Based on the results obtained at lab scale and under the conditions of the experiments herein, the following conclusions could be made:

- The feeding speed is the most significant factor for the UV lacquer curing;
- At slow feeding speed of the details ( $U=1\div 3$  m/min), the UV curing could result in considerable production defects;
- The quantity of applied varnish does not influence much on the UV curing time but has big impact on its adhesion.

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## Assessing hardwood flows from resource to production through Material Flow Analysis

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**Keywords:** Material Flow Analysis, hardwood, wood-based bioeconomy, wood resource potential, Sankey-diagram, hardwood resource, hardwood resource assessment.

### ABSTRACT

Many doubts accompany both the resource potential of hardwoods and their softwood substituting potential within its material utilisation. However, as the feedstock demand will rise with more and more wood-based biorefineries entering the market, wood supply security is an increasingly pressing topic. Currently, hardwood flow models are lacking, but are an essential basis for estimating future hardwood supply security. This study investigates the actual hardwood flows in Germany by applying a Material Flow Analysis (MFA). Visualising the results of the MFA for the reference year 2016. The MFA displays 4.44 +/- 0.12 million m<sup>3</sup> (fob) recorded fellings in privately-owned forests, 8.60 +/- 0.15 million m<sup>3</sup> (fob) recorded fellings in state forests, 9.31 +/- 0.52 million m<sup>3</sup> (fob) as unrecorded harvest from forests (i.e. 37% of the total harvest) and 2.65 +/- 0.35 million m<sup>3</sup> (fob) unrecorded fellings from non-forest area (11% of total harvest). Of that, almost 30% were used for materials and 70% were used for energy. Consequently, the potential for material use of hardwood in Germany lies beyond its current usage, especially given that good qualities are being used for energy due to actual low material consumer demand. Thus, the MFA assessed the quality and quantity of the German hardwood potential and showed that it offers significant possibilities for value added usage options such as, for example, producing innovative products in biorefineries using degraded high-quality assortments. Due to the low data consistency, heterogeneous datasets for the forest and wood industry sector were calibrated through data reconciliation and error propagation. Additionally, the lack of statistical data for specific wood industry sectors raised a problem, for instance, actual data on private combustion of wood, established hardwood uses, or new innovative products. Severe data issues regarding the German hardwood flow were confirmed.

### INTRODUCTION

Wood is currently hyped as one of the most important renewable sources for the bioeconomy currently being established in Europe. In terms of climate change mitigation, sustainable utilisation of wood is a central topic in strengthening circular economy (EC 2019; Toppinen et al. 2020; Winkel 2017). In Germany, hardwood forests are indigenous and offer a potentially abundant wood supply, which is increasing as a result of forest conservation measures (BMEL 2017a). Material utilisation of hardwoods offers a significant contribution to climate mitigation since hardwoods are currently being mainly used for energy purposes (Bösch et al. 2015; Jochem et al. 2015; Mantau 2018, 2012b; Weimar 2011). Lenglet et al. (2017) show the paradox of an abundant resource hardwood in France, but final value-adding processes usually taking place abroad. However, research approaches for the possible use of hardwood, especially beech wood, is already widespread in the wood building sector research (e.g. Auer et al. 2016; Hartig et al. 2016) and, in the near future, hardwood is predicted to be increasingly utilised as raw material for wood-based biorefinery systems (Duetsch 2019; Neupane et al. 2011; Teischinger 2019; UPM 30.01.2020). However, there are still uncertainties about the volume of hardwood available for the market (Mantau 2018). Studies investigating industrial scale (hard)wood-based biorefinery concepts in Germany (Michels 2014, 2009; Nitzsche et al. 2016) provide contradictory results on both the hardwood which is potentially available for the market and its probable contribution to the fulfilment of demand.

Available studies for Germany mainly focusing on softwood or just on parts of the hardwood network (FNR 2018; Mantau 2015, 2012b; Sörgel und Mantau 2006; Weimar et al. 2012) and are mainly based on primary data surveys. The applied methods provide a first overview, but are affected by missing data, unresolved data contradiction, the exclusion of important assortments (e.g. bark, sawmill by-products) and do not consider the entire hardwood network. As feedstock demand will rise (Jong et al. 2012) and with more and more wood-based biorefineries entering the market, which additionally boost this trend, wood supply security is a pressing topic. Therefore, a systematic view of the whole system including all sectors of the hardwood network is of increasing importance (Mantau 2015; Weimar 2011). As of yet, such integrated hardwood network models are lacking, but are seen as an essential basis for estimating future hardwood supply security.

A systematic review of the entire hardwood network including all hardwood-based industries is of increasing importance (Budzinski et al. 2017; Mantau 2015; Weimar 2011). Material flow analysis (MFA) is a suitable method for systematically documenting material flows in a defined space and time (Cencic und Rechberger 2008) and provides the means for reconciliation of heterogeneous data (Lenglet et al. 2017). The MFA method was chosen since it allows the incorporation of the highly inconsistent hardwood data sets which were observed. The aim of this study was to systematically document the entire hardwood flow network in Germany by applying an MFA. Accordingly, hardwood flows are assessed and reviewed starting with the supply volume at the forest and ending with the volume of hardwood processed in the different industry sectors or, used for energy purpose, supplemented by import and export flows (Figure 1).

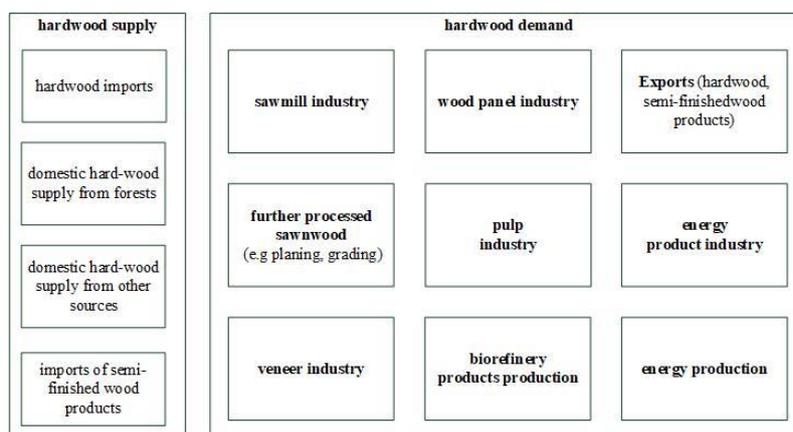


Figure 1: The hardwood supply and demand network focussed in this study

## METHOD AND MATERIAL

### System boundaries

Figure 1 provides an overview of the sectors in the German hardwood flow network and the system boundaries within this study. The consumption of semi-finished hardwood products (e.g., plywood within plywood panels, further processed sawnwood, i.e. planed, graded, finger-jointed) and the consumption of finished products by consumers, e.g. private households were excluded. The temporal boundary of the Material Flow Analysis (MFA) was one year because required data are mainly recorded yearly (TU Wien IWR 2012). Even though the spatial boundary is Germany, import and export flows are considered in accordance with the assortments, with by-products and semi-finished products integrated in the MFA. The time unit of the MFA is one year since required data are mainly recorded annually.

### Units and Data

Wood assortments and (semi-) finished wood products are often measured in different units as wood markets show a high variety within their measuring systems (Mantau 2015; Weimar 2011). Two reference units are common within wood sector analysis: solid wood equivalent (swe [ $\text{m}^3$  (s)]) (Mantau 2015) and wood fibre equivalent ( $\text{m}^3$  (f)) (Weimar 2011). The use of wood fibre equivalent is the more appropriate for MFA (Lenglet et al. 2017). Based on the wood fibre equivalent, a downstream calculation of by-products

is possible, e.g., by-products are counted as results of the sawing process and can be allocated to following processes separately (Bösch et al., 2015). Thus, wood fibre equivalent (m<sup>3</sup>(f)) was chosen as the reference unit. It describes the volume unit and the equivalent volume of wood fibres or wood-based fibres in the fibre-saturated state contained in the product. Since the MFA considers bark as a forest resource it is included in the data. Consequently, the unit of fibre equivalent over bark (fob) is used in this study. Conversion factors that were applied in calculations are listed in Table 1. The main data sources available were evaluated for information content, data reliability, wood assortments specifications and product codes (Table 2).

**Table 1: Conversion factors (Weimar, 2011)**

<i>product</i>	<i>unit</i>	<i>conversion factor for m<sup>3</sup> (f)</i>
roundwood	m <sup>3</sup> under bark	1.00
fuelwood (firewood, wood chips)	m <sup>3</sup> under bark	1.00
garden wood	m <sup>3</sup> under bark	1.00
by-products / processing residues	m <sup>3</sup>	1.00
hardwood by-products / processing residues	t air dry	1.42
post-consumer wood	t air dry	1.82
sawn wood	m <sup>3</sup>	1.00
Veneer	m <sup>3</sup>	1.00
Plywood	m <sup>3</sup>	0.96
particleboard	m <sup>3</sup>	1.25
MDF	m <sup>3</sup>	1.39
fibre boards	m <sup>3</sup>	1.47
chemical pulp	t	2.13
wood pellets	t	2.22
wood briquettes	t	2.22
charcoal	t	1.65

**Table 2: Data availability, data reliability and used data**

<b>report or statistics</b>	<b>information included</b>	<b>reliability</b>
wood market report of the German Federal Ministry of Food and Agriculture (BMEL 2017b, 2016, 2015, 2014; BMELV 2013, 2012, 2011, 2010, 2009, 2008)	wood harvest, producer price index, production volume of wood products, imports and export, etc.	<ul style="list-style-type: none"> <li>- wood harvest: underestimation because private forest owners have no reporting obligation</li> <li>- no differentiation between hardwood assortments within import and export</li> <li>- no differentiation between soft- and hardwood within import and export of wood fuels and by-products</li> </ul>
production statistics of the German Federal Statistical Office	Production volume of hardwood based-products	<ul style="list-style-type: none"> <li>- underestimation due to cut-off threshold by company size (employee or/and turnover)</li> <li>- not every reporting company is reporting every year, production volumes are missing or not listed</li> </ul>
working paper of raw wood and semi-finished wood products of the German Federal Statistical Office	consumption, stocks, production volume of German sawmill, veneer, plywood and wood panel industry	<ul style="list-style-type: none"> <li>- full survey with cut-off threshold by company size (employee or/and turnover)</li> <li>- inaccuracy due to the cut-off threshold and missing answers (e.g., too late)</li> <li>- within some years consumption and production volumes are missing or not listed</li> </ul>
statistical annual report of the German Federal Ministry of Food and Agriculture	wood consumption of wood processing industries, production volume of wood products, imports and export, etc.	<ul style="list-style-type: none"> <li>- non-continuous data, e.g., only the years 2005, 2010, 2012, 2014-2017 are listed</li> <li>- missing data (within listed years)</li> </ul>
energy statistics of the German Federal Statistical Office	electricity and heat production	<ul style="list-style-type: none"> <li>- renewable sources are only separated in biogenic solid fuels</li> <li>- no separate differentiation of wood share within energy production</li> </ul>

**Table 2: Data availability, data reliability and used data (continued)**

report or statistics	information included	reliability
studies and fuel surveys of Döring et al. (2016), Döring et al. (2018a), Döring et al. (2018b) and Mantau (2012a)	consumption of wood in energy production	<ul style="list-style-type: none"> <li>- complete surveys of wood consumption within energy production in heat and power stations and in private households</li> <li>- partial differentiation between softwood and hardwood</li> <li>- iterations approximately every five to ten years, but not in the same year for private households and heat and power plants</li> </ul>
Forestry Production and Trade (FAOSTAT 2018)	wood production by assortments, wood product production and trade, statistical report generated by a database query	<ul style="list-style-type: none"> <li>- wide range of countries, values, specifications and years</li> <li>- no differentiation between hardwood assortments within import and export</li> <li>- no differentiation between soft- and hardwood within import and export of wood fuels and by-products</li> <li>- partly different forestry and wood product codes compared to the wood market report or the official production statistics</li> </ul>
wood resource monitoring Mantau et al. (2007), Sörgel und Mantau (2006), Mantau (2012a), Mantau (2012b), Döring und Mantau (2012), Weimar et al. (2012), Döring et al. (2016), Döring et al. (2017a), Döring et al. (2017b), Döring et al. (2017c), Döring et al. (2018b), Döring et al. (2018a), Zimmermann et al. (2018)	wood flow analysis and wood resource balances for Germany	<ul style="list-style-type: none"> <li>- complete survey for the wood based sector as single branch studies</li> <li>- estimations of wood production volumes below the cut-off thresholds</li> <li>- iterations approximately every five years, but not in the same year for every branch</li> <li>- partial differentiation between softwood and hardwood</li> <li>- no separate investigation of the veneer industry</li> <li>- wood resource balances include all wood species, for some sectors a differentiation in soft- and hardwood</li> </ul>
Annual report of the German Pulp and Paper Association (VDP)	pulp and paper production, used raw materials	<ul style="list-style-type: none"> <li>- underestimation due to cut-off threshold by company size (employee or/and turnover) within the official production statistics</li> <li>- members of the VDP are reportable to the association</li> </ul>
Production reports of the German fuel wood and pellet association (DEPV)	production, consumption, import and export of pellets	<ul style="list-style-type: none"> <li>- underestimation due to cut-off threshold by company size (employee or/and turnover) within the official production statistics</li> <li>- members of the DEPV are reportable to the association</li> </ul>

### **Methodological approach**

The methodological approach in terms of the basic principles of the material flow design and the standard unit (i.e., wood fibre equivalent) was developed by Weimar (2011) and from the applied approach of Lenglet et al. (2017), for the MFA of the hardwood supply network. In the first step, input and (by-) product output figures were documented mainly using data provided by the Federal Statistical Office of Germany (StBA). Hardwood supply, processing, and consumption sectors were then reviewed in relation to qualitative, quantitative, and technical parameters as the second step. If no official or scientifically published data were available or not sufficiently reliable, other data sources (e.g., wood monitoring and consumption studies, reports of federations, etc.) were used to close data gaps. In cases of entirely missing data, the authors had to make their own calculations based on assumptions. Following this, the MFA was performed by setting up the hardwood flow network connecting wood supplying and demanding sectors to each other. The MFA was conducted with the software tool STAN (TU Wien IWR 2012).

## RESULTS AND DISCUSSION

Showing the flow of hardwoods through the network, the MFA systematically compromises sources and sinks of hardwood network in Germany. Data uncertainties were resolved by data reconciliation and error propagation, since the available data sets are highly heterogeneous. MFA displays 4.44 +/- 0.12 million m<sup>3</sup> (fob) recorded fellings in privately-owned forests, 8.60 +/- 0.15 million m<sup>3</sup> (fob) recorded fellings in state forests, 9.31 +/- 0.52 million m<sup>3</sup> (fob) as unrecorded harvest from forests (i.e. 37% of the total harvest) and 2.65 +/- 0.35 million m<sup>3</sup> (fob) unrecorded fellings from non-forest area (11% of total harvest). Of that, almost 30% were used for materials and 70% were used for energy in 2016. MFA results in a hardwood volume from forests of 22.87 million m<sup>3</sup> (f) and by-products of 1.69 million m<sup>3</sup> (f). In all, 21.53 +/- 0.56 million m<sup>3</sup> (f) was used for energy, of which 14.06 +/- 0.51 (65%) million m<sup>3</sup> (f) was used in private households, 5.04 +/- 0.18 million m<sup>3</sup> (f) (23%) in energy plants > 1 MW, and 2.44 +/- 0.15 million m<sup>3</sup> (f) (11%) in energy plants < 1 MW.

MFA results (e.g. for veneer, panel, pulp, energy product and energy production industries) are mainly consistent with former studies (Bösch et al. 2015; Jochem et al. 2015; Mantau et al. 2016; Mantau 2015; Seintsch 2011; Seintsch und Weimar 2013; Weimar 2011), especially the considerable volume of unrecorded wood harvested in Germany, especially within hardwoods, where almost half of the total hardwood wood fuel harvest is unrecorded (4.23 +/- 0.24 million m<sup>3</sup> (f), 45%). Calculated assortment distribution of 73% wood fuel (including firelogs from forests which are used in private households) and 27% forest wood chips are in line with a Bavarian fuelwood study indicating a share of wood fuel/firelogs of 73% and forest wood chips of 27% (Gaggermeier et al. 2014). Demand for sawlogs/veneer logs by the sawmill industry was calculated to be 3.09 +/- 0.17 million m<sup>3</sup> (f) in 2016, which slightly exceeds the 2.73 million m<sup>3</sup> (fob) that was calculated based on the correction factor of 2.81 (Döring und Mantau 2012). Significant underestimation of sawmill production volumes by the official records resulting from both non-reporting from about 45% of companies above the reporting threshold (Zimmermann et al. 2018) and the high number of hardwood sawmills below the reporting threshold was confirmed by MFA. As energy production statistics list renewable sources aggregated only as biogenic solid fuels, hardwood demand for the energy production sector in Germany is hard to estimate. Therefore, more detailed information for energy production based on wood is needed. Approximately 74% (21.53 +/- 0.56 million m<sup>3</sup> (f)) of total hardwood (29.05 +/- 0.59 million m<sup>3</sup> (f)) is used in energy production, of which 12.78 million m<sup>3</sup> (fob) (44%) of fresh fibre hardwood from forests is directly used for energy purpose.

With no biorefinery producing on an industrial scale in 2016, MFA results just provide some indications for the potential coverage of future demand of this sector. A hardwood demand of 400,000 t (db)/a (0.851 million m<sup>3</sup> (f)) is assumed for a lignocellulose biorefinery located in Germany (Michels 2014, 2009; Nitzsche et al. 2016). With a detected oversupply of 2.08 million m<sup>3</sup> (f) (sawlogs/veneer logs) and 3.13 million m<sup>3</sup> (f) (pulpwood) currently used for energy production, the future demand of biorefineries could be satisfied. Nitzsche et al. (2016) showed that a biorefinery can supply energy as side flow to partly address this problem.

Heterogeneous datasets for the forest and wood industry sector were calibrated through data reconciliation and error propagation due to the low data consistency within the wood supply network. Lack of statistical data for specific wood industry sectors raised a problem (e.g. actual data on private combustion of wood or established hardwood uses and some new innovative products) and accordingly, results of the study confirm severe data issues regarding the German hardwood flow. MFA proved to be a suitable method to investigate hardwood flows characterised by highly heterogeneous data and facilitated a systematic investigation of the German hardwood network, revealing important insights on the forest and wood sector.

## CONCLUSION

An MFA systematically compiling sources and utilisations in the German hardwood network, shows the flow of hardwoods throughout the network. Since available data is highly heterogeneous, data uncertainties were resolved by data reconciliation and error propagation. Accordingly, hardwood flows are evaluated starting with the supply volume at the forest and ending with the volume of hardwood processed in the different industries, supplemented by imports and exports. Finally, research gaps and future aspects are discussed, concluding with the need for further investigation into the utilisation possibilities of hardwoods.

An MFA allows a more detailed view on the interlinking flows inside a demand and supply network as well as additionally solving data contradictions. Distinguishing between softwood and hardwood as well as assortments and sources contributes to forestry and the wood-based sector by providing appropriate monitoring in times of forest reconstruction due to climate change and by providing information for investment decisions requiring long-term planning and supply security.

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## Effect of alkaline treatment on bond strength and solid content of bio-based adhesives for plywood

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**Keywords:** Protein adhesives; bond strength; renewable sources; alkaline hydrolysis; protein denaturation; viscosity; solid content

### ABSTRACT

In this work the effect of alkaline hydrolysis on different plant proteins was studied to evaluate their wood bonding performance on beech plywood. The aim of comparing different plant proteins was to find alternatives sufficient available in Europe with comparable bonding performance to soy. Conditions of alkaline hydrolysis differed in temperature and NaOH molarity. During this study main investigated parameters were related to raw material itself, its water dispersion and its interaction with beech veneer. The relation between protein viscosity, solids content, solubility and bonding performance of alkaline protein dispersions was investigated. Tensile shear strength measurements were performed on beech wood substrates bonded with protein dispersions to investigate bonding performance. It was found that potato protein has competitive adhesive properties compared to soy, and was additionally able to form a dispersion with a high solid content at comparably low viscosity. Potato protein alkaline dispersions with different solid content (20 and 50%) were tested for dry and wet bonding performance. It was found that the potato protein suspension treated under 1M NaOH, then hot pressed at 140 °C performs tensile shear strength up to 7.5 MPa in dry condition, representing wood failure, and up to 0.7 MPa after water soaking for 24 hours.

### INTRODUCTION

Adhesives like urea-formaldehyde, are widely used in plywood, composite wood panels and furniture production because of their high adhesion strength and low cost. However, these adhesives are mainly derived from limited and non-renewable fossil sources and cause a negative impact on the environment. Some of those adhesives, especially urea-formaldehyde resins, may be able to emit formaldehyde. Due to increasing environmental concerns, adhesives based on proteins are becoming more attractive since they are derived from renewable resources. Plant proteins, such as soybean protein or wheat gluten, belong to a group of renewable biopolymers and are regarded as attractive due to their environmental friendliness and sustainability. In this work plant proteins produced in Europe such as wheat, potato, and pea proteins were compared with soy flour to study bonding performance, balance between solid content and viscosity and relation of bonding performance and solubility. Selected raw materials mainly consist of storage proteins, which differ in their structure and fractions content, which exhibit different properties. As a first step to increase their bonding properties, they need to be unfolded. There are different methods for protein denaturation, such as thermal (Mekonnen et al., 2013b; Pan et al., 2005), chemical and mechanical methods (Mekonnen et al., 2013a). The methods of chemical denaturation and hydrolysis for protein include alkali, surfactants, and organic solvents. Sodium hydroxide has been used extensively to denature various sources of proteins including soy protein (Hettiarachchy et al., 1995) and wheat gluten (Nordqvist et al., 2010;

Nordqvist et al., 2013).. NaOH treatment of protein enhances the adhesive property by exposing specific functional groups as a result of unfolding of protein molecules, and increasing the intermolecular interactions with the solvent/medium and reducing the viscosity (He, 2017). Soy flour denatured with strong alkali provided good plywood bonds and allowed the commercial production of the interior plywood (Lambuth, 1994). Although most of work was focusing on the development of adhesive systems with sufficient bonding strength, a systematic investigation to identify parameters influencing bonding characteristics is still missing.

Selected different raw materials have been hydrolyzed under different alkaline conditions and temperature to study its influence on bonding performance. Potato protein as a domestic raw material was tested for wet bonding strength with different solid content. Results obtained for dry and wet bonding performance are groundwork for further research.

## EXPERIMENTAL METHODS

### **Materials**

Commercial proteins used in this work were kindly provided by Agrana Research and Innovation Center GmbH (Tulln-an-der-Donau, Austria) – potato protein (80±0.6% total protein content), wheat gluten (84±0.7% total protein content); Emsland-Starke GmbH (Emlichheim, Germany) – pea protein (78.2±0.3% total protein content); Cargill Deutschland GmbH (Krefeld, Germany) – soy flour (52.7±1.2% total protein content).

### **Alkaline hydrolysis**

Aqueous sodium hydroxide solutions (0.1 M and 1M) were used as dispersing agents. The protein samples were added while stirring at room temperature and then the desired temperature was adjusted with the deviation +/-3°C. Vacuum distillation was subsequently used to remove excess water from the protein dispersions to reach higher solid content. Selected dispersions were distilled under vacuum at a maximum temperature of 40 °C under constant stirring to retain homogeneous mixture. The amount of removed water was measured by collecting the water condensate. Solid content and viscosity were tracked until the viscosity reached a level which was considered to be too high for applying during wood composites production. Solid content of the dispersions was measured by oven drying at 103 °C with samples of 3g during 1 hour according to ISO Standard 3251. Results indicated for each measurement were calculated as average from 3 samples. For the viscosity measurement a rheometer from Bohlin Instruments Ltd (Bohlin CVO 50, Bohlin, Cirencester, United Kingdom) with cone-plate set up 4° of 40 mm diameter was used. The temperature level of the instrument was set to 20°C, the gap size 150 µm and a constant shear rate of 200 s<sup>-1</sup> were selected. The applied amount of sample was 0.7 ml. During 2,5 minutes, 10 measurements of the viscosity were done and the average of them was calculated.

### **Tensile shear strength measurement**

For bonding strength measurement an Automated Bonding Evaluation System (ABES, Adhesive Evaluation Systems, Inc., USA) was used for forming and breaking the bonds to determine strength and wood failure. Measurements were evaluated according to the corresponding ASTM D7998-15 (ASTM International 2015). Beech veneer samples with the dimensions 117 x 20 x 0.6-0.8 mm<sup>3</sup> preliminary stored in standard climate (20°C, 65 % RH) were used as adherend stripes. A spread rate of 150 g/m<sup>2</sup> (dry matter) for each adhesive suspension was applied, calculated based on precisely determined solid content. For dry bonding strength a required amount of adhesive was added to a 5 mm wide overlapping area at one end of one sample with subsequent hot pressing for 10 minutes. For wet bonding strength hot pressed samples soaked in water at room temperature for 24 hours. For each protein suspension five samples were measured.

### **Solubility characterization**

Treated and untreated protein dispersions in amounts of 0.1 g were neutralized, then centrifuged at 10000 rpm for 15 minutes and the precipitate was diluted 100 times with water. Diluted dispersions were stirred during 1 hour at ambient temperature and again centrifuged at 10000 rpm for 15 min at 5 °C. Supernatant protein content was determined by Bradford Assay with a Fluorescence Spectrophotometer (F-7100, Hitachi, Japan) at a wave length of 595 nm. Roti-Quant (Carl Roth, Germany) was used as colorimetric

reagent for detecting and quantifying soluble proteins. Bovine serum albumin (BSA) was used as standard (Sigma Chemical Co., USA). Protein solubility was expressed as:

$$\text{Solubility (\%)} = \frac{\text{Protein in the supernatant (mg/mL)} \times 100}{\text{Initial protein in the sample (mg/mL)}} \quad (1)$$

## RESULTS AND DISCUSSION

Solubility values of dispersions are shown in Fig. 1. All proteins show an increase of solubility with increase of NaOH molarity. Potato protein is the most soluble in this case due to its molecular structure (Zhang et al., 2016). Temperature effect is less pronounced compared to alkaline concentration.

Fig. 2 represents the results from the tensile shear strength measurements using a solid content of 25%. NaOH treatment of protein enhances the adhesive property by exposing specific functional groups as a result of unfolding of protein molecules, and increasing the intermolecular interactions with the solvent/medium and reducing the viscosity (Hettiarachchy et al., 1995). Potato and soy protein showed highest values of tensile shear strength after treatment under 1M NaOH at 60°C. At these conditions the highest solubility values were obtained for these protein samples.

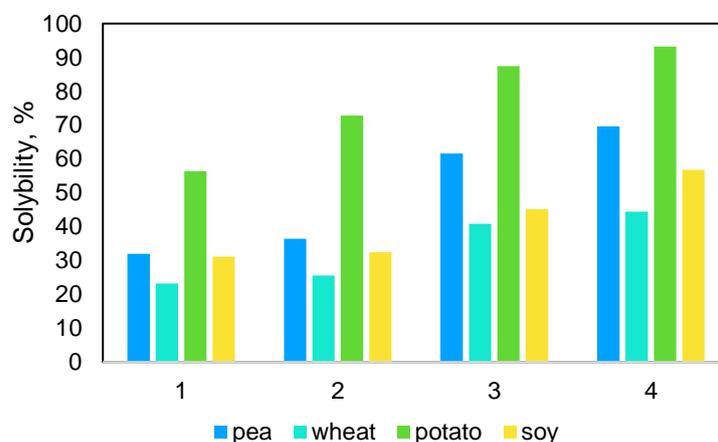


Figure 1: Solubility of protein dispersions treat under conditions: 1 – 0,1M NaOH 30°C, 2 – 0,1M NaOH 60°C, 3 – 1M NaOH 30°C, 4 - 1M NaOH 60°C

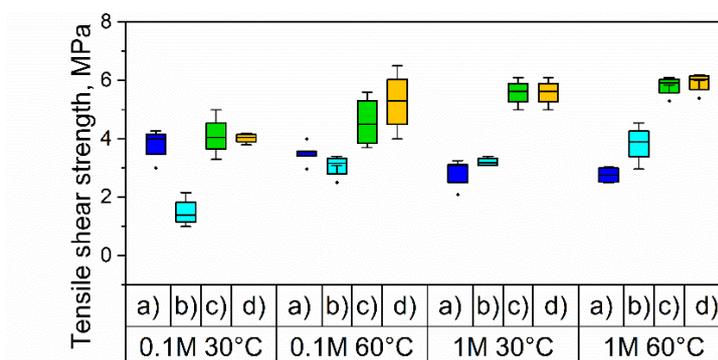


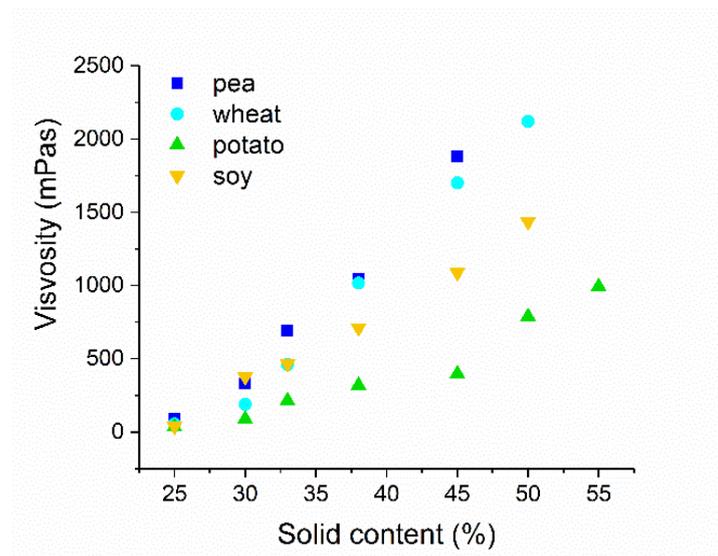
Figure 2: Tensile shear strength of protein adhesives after hot pressing for 10 min at 110°C temperature of press, measured on an Automated Bonding Evaluation System (ABES). a) pea, b) wheat, c) potato, d) soy

One of the benefits of potato protein, which showed better bonding performance similar to soy protein in this case, is probably its solubility and therefore the ability to form a homogeneous system. Comparing to other proteins, wheat gluten mostly consists of water insoluble gliadins and glutelins, linked with disulfide bridges, which impeded penetration of its structure into the wood surface. By reducing disulfide bonds

under 1M NaOH an increase of tensile shear strength was also observed for wheat gluten. Generally, adhesives with high viscosity do not flow easily on the surface and into the pores, resulting in insufficient bond strength, while adhesives with too low viscosity lead to excessive penetration and bond line starvation with subsequent decreasing bonding properties (Bacigalupe et al., 2015; Nordqvist et al., 2010).

To achieve a proper adhesion, the adhesive must have the capacity to sufficiently flow, wet and penetrate into the rough surface of the substrate, in order to work as an anchor, while maintaining sufficient adhesive in the bond line. Adhesive penetration is generally believed to have a strong influence on bonding performance. Adequate penetration provides a substantial interphase that promotes interaction, perhaps reaction, and also mechanical interlocking. On the other hand, excessive penetration could lead to a “starved” bond-line having poor performance (Gardner et al., 2014; He, 2017).

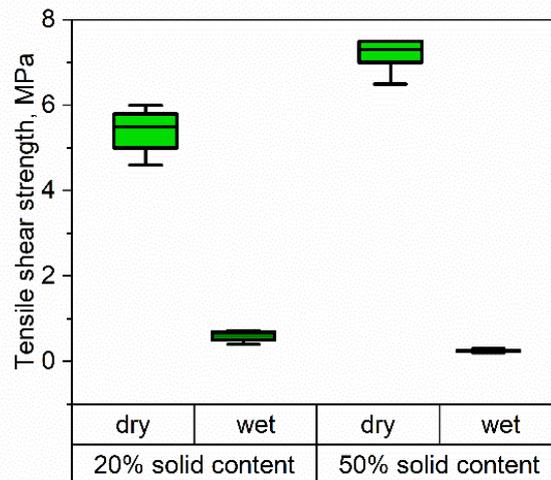
Protein dispersions treated under 1M NaOH at 60°C have been selected for solid content increasing experiment by vacuum distillation because of its lowest viscosity and better homogeneity. All four dispersions of protein hydrolysates exhibit quite smooth increase of viscosity with increasing of solid content (Fig. 3). Potato protein showed a less intensive increase of viscosity and higher solid content than the others. Other proteins seem to have more pronounced increment of viscosity at solid content above 30%.



**Figure 3: Viscosity change of protein alkaline hydrolysates with increasing solid contents**

Low solid content is a major problem for the production of composites, such as particleboard and fiberboard. The excess water cause steam pressure during production, wood swelling and may result in considerable stresses. The balance between viscosity and solids content is one of the biggest issues in order to get high bond strength and spread easiness of the adhesive and thereby improve its ability to properly wet, flow, and penetrate into the wood substrate (Frihart and Lorenz, 2019; Frihart and Satori, 2013).

Potato protein as a raw material performed comparable to soy with regard to dry bonding strength. The effect of increased solid content was tested for both dry and wet bonding strength (Fig.4).



**Figure 4:** Tensile shear strength of potato protein adhesives with different solid content after hot pressing for 5 min at 140°C temperature of press (dry) with subsequent soaking in water for 24 hours at ambient temperature (wet) measured on an Automated Bonding Evaluation System (ABES).

The results of tensile shear strength for dry bonding are higher at 50% solid content. In this case for all samples wood failure from 50 to 100% was observed. The results of tensile shear strength for wet bonding after 24 hours of soaking in water at ambient temperature are slightly higher for adhesive with 20% solid content, which has lower viscosity and probably penetrates deeper into wood substrate surface.

## CONCLUSIONS

Different plant proteins including soy protein as a reference were exposed to alkaline hydrolysis and their effect on viscosity, solubility and bonding performance was observed. The results showed improved bonding performance as an effect of the different treatments, with superior result using 1M NaOH at 60 °C for potato and soy proteins. Soy and potato proteins performed highest tensile shear strength after treating in this condition with average values up 5.75 MPa in dry testing conditions. An increase of bonding performance is also correlating with an increase of solubility this protein. Investigating the effect of increasing the solid content of treated dispersions was performed via vacuum distillation with the aim of improving the balance between solid content and viscosity. Potato protein showed higher solid content compared to other proteins (around 55%) at a moderate target level of viscosity of 1000 mPa s. This is also explained by higher solubility of potato protein and ability to form homogeneous system. For soy a similar viscosity level was already reached at approx. 42% solid content, and much lower levels for the other proteins. Wet bonding performance testing showed that potato protein alkaline suspension performs tensile shear strength up to 0.7 MPa after hot pressing at 140 °C temperature for 5 min and soaking in water for 24 hours. Increased solid content did not show significant influence on bonding performance.

This study showed that alkali modification is a suitable procedure to modify protein suspensions to improve the dry and wet bonding behavior of wood substrates, while industrially relevant processing properties of the adhesive can be reached.

## ACKNOWLEDGEMENTS

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## The first results of shrinkage and swelling of lactic acid-treated beech wood

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**Keywords:** impregnation, polymerization, solid wood, dimensional stability, HPLC, acetic acid, furfural

### ABSTRACT

Lactic acid (LA) is an organic acid that can be found in nature. Polymerization of LA produces poly-lactic acid (PLA), which is classified as thermoplastic. As an example, it is used to produce biodegradable packaging materials. In addition, PLA can play an important role in improving the properties of wood, such as dimensional stability. Several methods for producing PLA are already known and the impregnation of some wood species with LA is partially solved as well. These processes influenced positively many wood properties, but still many questions remained to be unanswered. In this study the heat pre-treated (oligomerized) LA were impregnated in the wood tissue by vacuum treatment, followed with a heat treatment on 120 °C for 6 hours to achieve the polymerization of lactic acid. The shrinkage and swelling of beech wood were examined after performing an impregnation process with LA, followed by a polymerization process.

First, the dry specimens were saturated with distilled water. The LA impregnation and polymerization treatment resulted in a very low swelling of wood in every anatomical direction (Tangential, Radial and Longitudinal; T, R and L), compared to untreated specimens. However, during the drying cycle, the shrinkage of treated wood highly increased compared to its swelling. This may be attributed to the leaching of the LA that was not well polymerized. Of course, the shrinkage of treated specimens still did not reach the shrinkage of untreated wood.

The chemical composition of wood samples by high-performance liquid chromatography (HPLC) revealed that PLA-treated wood had elevated furfural, hydroxymethyl furfural and 5-methyl furfural levels compared to untreated wood, which indicated slight decomposition of the hemicelluloses. After PLA treatment the samples had a weight gain of 765 mg/g. Unfortunately, PLA treated wood had a free lactic acid content of about 90 mg/g (related to absolute dry wood) which confirmed that under the applied treatment conditions not all of the lactic acid got polymerized.

### INTRODUCTION

In general, wood have significant lateral shrinkage and swelling. This phenomenon causes serious problems in the use of wood. As its moisture content changes, so behave its physical-mechanical properties, including the dimensions. Because the dimensions vary differently in the 3 main anatomical directions of the wood, it can be a source of serious deformations. Typically, the properties vary from the absolute dry state to the fiber saturation point. The change ratio is about 10% in the tangential direction and about 4% in the radial direction. In the longitudinal direction, natural wood has minimal dimensional change, a few tenths of a percent. However, some treatments, e.g. longitudinal wood compression (parallel to the fiber direction) significantly increases the longitudinal shrinkage and swelling. Thus, reducing shrinkage-swelling is an important issue in many areas of wood science.

Researchers have long been concerned with reducing shrinkage-swelling of wood and with the resulting anisotropy-reduction. Many methods have been developed, such as heat treatment in different atmospheres, saturation with different materials, chemical treatments, and countless combinations thereof. Unfortunately, all of them have drawbacks, the result cannot be uniformly positive in all respects. In many cases, in addition to the improvement of dimensional stability, there is a significant deterioration of the mechanical properties or the natural wood becoming a polluting composite, which causes serious problems in the further use of the product. In our study, we have been thinking of a solution that is not expected to significantly impair the mechanical properties and preserve the environmentally friendly nature of wooden materials. Finally, we got to the application of impregnation wood with lactic acid (LA).

LA is a colorless, liquid organic compound that can be inexpensively produced in large quantities from materials with high starch content, such as corn. Its formula is  $C_3H_6O_3$ . Two main variants are known, L-lactic acid and D-lactic acid, which have the same properties but differ in their optical rotation direction. It is already used for a countless of purposes in various industries, just think of plastic cutlery, bags and sacks known as eco-friendly, or the filaments of 3D printers. It can be found in the textile and clothing industry and is also used in the food and beauty industries (Bodnár 2002, Chahal and Starr 2006). Material that can be produced in large quantities at low cost (Hetényi 2010). Its good usability and eco-friendly nature lies in the fact that it behaves like a plastic during use, but after it becomes waste, it decomposes naturally under the right conditions. This requires proper temperature, humidity and presence of bacteria.

LA molecules are extremely hydrophilic, but when polymerized, the compound stabilizes, and the long molecular chains are only able to bind a minimal amount of water (poly-lactic acid; PLA). If the polymerization takes place in solid wood, the polymer chains filling the cell lumens can prevent or significantly reduce the penetration of water molecules into the wood. It is a difficulty that many wood species cannot be properly impregnated even with low molecular-size materials due to their structure (e.g. oak, robinia). It is also a problem that water is formed during the polymerization, which highly influences the wood materials as previously described. Using an impregnation material with long polymer chains, easily saturable wood species (e.g. beech, poplar) cannot be handled properly either, so the polymerization of LA must be carried out inside the wood.

The polymerization of lactic acid molecules naturally takes place very slowly with poor efficiency. In industrial processes, the catalysts (e.g. low amount of sulphuric acid or titanium butoxide or tin (II) 2-ethylhexanoate) and heat extremely accelerates the polymerization process (Noël et al. 2009). In addition to the duration of the polymerization, the resulting size of the PLA molecule is also very important for the quality of the PLA. The longer the molecular chains, the more stable and stronger the product. However, the addition of different chemical catalysts reduces the environmentally friendly nature of the final product, so we used a special cooking process in our experiments. In this case less long molecular chains are formed in a slightly longer time compared with the catalysation with chemicals, but we have tried to use a process that still gives adequate properties for PLA.

Both LA and PLA as a compound are already well known and the impregnation of some wood species with LA is partially solved as well. In earlier studies these processes influenced positively many wood properties, for example anti-swelling efficiency, equilibrium moisture content, moisture exclusion efficiency, significant biological resistance. Unfortunately, modulus of elasticity decreased by the treatment (Noël et al. 2015, Grosse et al. 2019), but still many questions remained to be unanswered. To decrease the amount of the remaining questions, we performed shrinkage-swelling tests on untreated and PLA-treated beech samples. We also wanted to know the changes in the chemical composition of wood as a result of the treatment, so we performed high-performance liquid chromatography (HPLC).

## EXPERIMENTAL METHODS

Experiments were performed using L(+)-lactic acid for its easier availability. The liquid, transparent 90% aqueous solution of L (+)-lactic acid from Acros Organics b.v.b.a. (Belgium) was used as impregnating raw material. Because of the LA monomer contained 10% water, it was first boiled under a vacuum of 150 mbar at 75 °C for 75 minutes to allow the water content to evaporate from the solution. During the whole curing period of LA, we used a heated magnetic stirrer at 175 RPM rotation speed. The dehydration was followed by an oligomerization process in two steps. The concentrated LA monomer was boiled under a vacuum of 150 mbar at 100 °C for 100 minutes and then the temperature was raised to 130 °C for another 160 minutes. When the vacuum was applied as well as when the temperature was raised, we made the changes carefully in small steps, taking care not to splash and boil out the LA from the beaker. At this time, the monomers oligomerized and, after cooling, this oligomerized material had a gel consistence. Presumably, in this state, the solution contained both monomers and oligomers. We stored it in a closed container protected from light until further use, it could not absorb water from the air.

Beech (*Fagus sylvatica* L.) wood species were used, altogether 3 samples per groups. The samples came from the heartwood section of one log and were sawn in the first step to dimensions 20×20×200 mm<sup>3</sup> (tangential × radial × longitudinal; T × R × L), paying attention to the three principle anatomical directions

of wood. The first group contained the untreated samples, while the second contained the LA-treated samples. After sample preparation, the moisture content (*MC*) of all samples were still over the fiber saturation point. All samples were climatized to 12% *MC*, then carefully dried in a Memmert 100-800 drying oven (Mettler-Toledo GmbH, Germany) to a final temperature of 103±2 °C to avoid cracks.

In the impregnation phase, the absolutely dry wood samples were placed in the LA oligomer and then heated in vacuum. The impregnation process was carried out at 100 mbar at 90 °C temperature for 60 minutes, followed by a sudden increase in pressure. After the impregnation process, the samples were wiped and wrapped in aluminium foil. The foiled samples were put in a heating chamber at 120 °C for 6 hours to polymerize the LA oligomers. When the polymerization process finished, the aluminium foil was left on the samples for cooling and placed in a desiccator so that they could not absorb water. Later the foil removing has been easily done.

After drying or impregnation of the samples, small specimens for the shrinkage-swelling tests were cut with dimensions of 12x12x20 mm<sup>3</sup> using a circular saw, paying attention to avoid moisturizing the specimens, and to be each surface freshly sawn. Both the dimensions in the three anatomical directions with a Helios 23004009 digital dial indicator (Helios-Preisser GmbH, Germany) and the weight of each specimens with a Precisa XT 1220M-FR scale (Precisa Instruments AG, Switzerland) were measured. Then all of the specimens were soaked in distilled water for 4 days and after soaking, their weight and dimensions were measured. After that, the specimens were carefully climatized, dried to 0% *MC* and measured again. Then the specimens were tested through one more cycle of soaking in distilled water until the saturated state and oven drying.

The *MC* relative to net dry weight, the swelling coefficient in the radial ( $\alpha_r$ ), tangential ( $\alpha_t$ ) and longitudinal ( $\alpha_l$ ) directions, volumetric swelling ( $\alpha_v$ ), the shrinkage coefficient in the radial ( $\beta_r$ ), tangential ( $\beta_t$ ) and longitudinal ( $\beta_l$ ) directions and the volumetric shrinkage ( $\beta_v$ ) were calculated as described in the ISO 13061 standard package (Eq. 1 and Eq. 2).

$$\alpha = (l_2 - l_1) / l_1 \quad (1)$$

$$\beta = (l_1 - l_2) / l_1 \quad (2)$$

where  $l_1$  is the saturated dimension of the test piece in mm and  $l_2$  is the oven-dry dimension of the test piece in mm. The anti-swelling efficiency ( $ASE_\alpha$ ) and the anti-shrinkage efficiency ( $ASE_\beta$ ) were calculated separately in the three main anatomical dimensions, using Eq. 3 and Eq. 4:

$$ASE_\alpha = \frac{\alpha_{ut} - \alpha_t}{\alpha_{ut}} \cdot 100 \quad (3)$$

$$ASE_\beta = \frac{\beta_{ut} - \beta_t}{\beta_{ut}} \cdot 100 \quad (4)$$

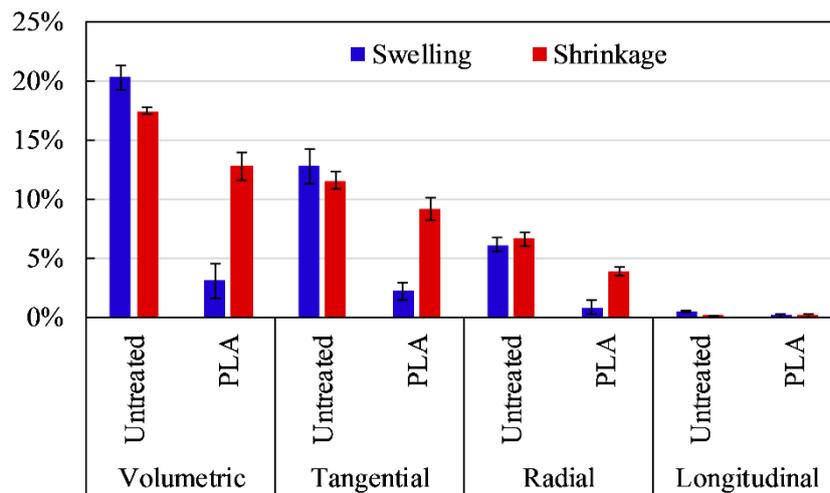
where  $\alpha_{ut}$ ,  $\beta_{ut}$  are the swelling coefficient and shrinkage coefficient, respectively, of untreated wood in % and  $\alpha_t$ ,  $\beta_t$  are the swelling coefficient and shrinkage coefficient, respectively, of treated wood in %.

The chemical composition of wood samples were obtained by high-performance liquid chromatographic (HPLC) separation (Shimadzu LC-20 equipment, Shimadzu Corporation, Kyoto, Japan) using a Bio-Rad Aminex HPX-87H column (Bio-Rad Laboratories Inc., Hercules, USA) at 60 °C, with 0.6 ml/min flow of 0.005 M H<sub>2</sub>SO<sub>4</sub> mobile phase, and UV detection at 210–240 nm. Wood chips were prepared both from untreated and LA-treated samples with room-dry (5-9%) *MC*, carefully separating the different sample types to avoid contamination. Wood samples were ground using a Retsch SK3 type grinder (Retsch GmbH, Haan, Germany), then sieved, resulting a grain of size 0.2-0.63 mm. For the extraction of acids 0.3 g sample was weighed in a 50 ml centrifuge tube (PP) and 30 ml distilled water was added. The mixture was extracted using an ultrasonic bath (Elma Transsonic T570, Elma Schmidbauer, Singen, Germany) for 3 x 10 minutes, keeping water bath temperature between 26-33 °C. The statistical analysis has been done using the Dell Statistica version 13 software (Dell Inc, USA). We tested the changes of the selected constituents as a result of the treatment. Fisher LSD test was used to evaluate the effect of the treatments on the investigated samples with a significance level of  $p < 0.05$ .

## RESULTS AND DISCUSSION

During impregnation, air bubbles were leaving the wood due to the vacuum. At the same time, much less LA remained in the tray in which the impregnation has been done. This predicted the success of the impregnation process. The fiber saturation point of the beech wood species is 35.6% (Báder and Németh 2019), so the absolute dry sample will swell under the effect of the first 35.6% water uptake. At this point, the cell walls reach their maximum size and their interfibrillar and intermicellar cavities expand as much as possible. Significant amounts of water – or in this case LA monomer and LA oligomer – enter and may form chemical bonds with the cell wall, or they can be fixed in the cavities of the cell wall by forming long, stable chains upon polymerization. Although LA was 100% dehydrated, it behaved as water and caused a very high swelling for the initially absolutely dry wood samples (13.7%, 4.6% and 0.2% in the anatomical directions T, R and L, respectively). Accordingly, the density of the samples increased averagely by 48% as a result of the impregnation-polymerization treatment. In addition to the time required for polymerization, time and opportunity must be provided for the newly formed water to also leave the wood. Water molecules are likely to form chemical bonds with hydrophilic wood and highly hydrophilic LA as well. But in this way, water inhibits the formation of wood-LA bonds as well as the conversion of LA molecules and oligomers into long chains. Thus, by removing the water, we can further aid in the best possible polymerization.

Shrinkage-swelling tests were performed on both untreated and impregnated specimens. The absolutely dry specimens were soaked in distilled water and then dried again, which gave quite different shrinkage-swelling results for the treated specimens (Fig. 1).



**Figure 1: Results of the swelling-shrinkage test of untreated beech wood and wood impregnated with PLA followed by a polymerization process, based on the average values of 6 measurements. The error bars show the standard deviations**

While the shrinkage and swelling of the untreated specimens were similar in either anatomical direction, only small swelling could be observed in the samples impregnated with LA. This may be due to the impregnation, because during the treatment the wood reached its near-maximum size and was only able to swell minimally with further water uptake. The water uptake of the untreated and treated specimens was averagely 88.4% and 34.8%, respectively. This is the result of the presence of LA in the cell lumens. However, during drying, there was a much greater shrinkage relative to swelling, although lower in all anatomical directions compared to the shrinkage of untreated specimens. This indicates a significant leaching of LA during soaking in the course of the swelling test. The difference in absolute dry masses before and after soaking shows that 31.4% from the 76.5% impregnation material originally impregnated into the wood was washed out. This amount of LA did not just come from cell lumens, but partially released from the cavities of the cell walls, allowing significant shrinkage of the specimens. However, the dimensional stability of the material has still improved volumetrically by 36%, which means 12.8% shrinkage instead of 17.4%. It can be stated that the polymerization treatment of the impregnated wood-LA

composite was not adequate. The heating of the impregnated samples will have to be done for a longer time later, which is likely result in larger polymer chains and stronger chemical bonds, thus, the soaking test will not have such a significant leaching and shrinkage effect on the specimens.

The changes of the selected components in our samples were examined by high-performance liquid chromatography (HPLC). As a result of the treatment with LA and heat, the amount of acetic acid increased slightly (from 1.16 to 1.34 mg/g), but the statistical analysis did not indicate this as a significant change. More conclusions can be drawn from the amounts of LA and furfural. The amount of furfural as well as 5-methyl furfural was not detectable in the untreated samples, but these constituents appeared in the samples treated with LA at 0.11 mg/g and 0.01 mg/g, respectively. The amount of hydroxymethyl furfural also increased significantly from 0.02 mg/g to 0.25 mg/g. The amount of LA monomers was 0.58 mg/g in the untreated samples and 89.73 mg/g in the treated samples. The items discussed are easily reviewed in Table 1.

**Table 1: The average HPLC test results followed by the standard deviations of untreated and lactic acid (LA)-treated wood. Component content is in mg/g**

	Acetic acid	Furfural	5-methyl furfural	Hydroxymethyl furfural	LA uptake	LA residue monomer
Untreated wood	1.16 ± 0.06	0.00 ± 0.00	0.00 ± 0.00	0.02 ± 0.00	0.00 ± 0.00	0.58 ± 0.02
LA-treated wood	1.34 ± 0.07	<u>0.11</u> ± 0.01	<u>0.01</u> ± 0.00	<u>0.25</u> ± 0.02	<u>765.01</u> ± 101	<u>89.73</u> ± 7.76

*Significant differences ( $p < 0.05$ ) are marked with underlined numbers*

The presence of LA monomers indicates that a significant amount of unoligomerized and unpolymerized LA remained, which may be easily leached during the shrinkage-swelling tests. Based on the weight gain during the impregnation with LA, a total of 765 mg/g of LA was added to the wood. Comparing these two values, it turns out that about 12% of the introduced LA was not even oligomerized. The presence and increase of furfurals are chemically not directly related to LA impregnation. These components are formed by the decomposition of hemicelluloses under the influence of the heat applied during treatment. Since very small amounts of furfurals could be detected, a slight degradation of hemicelluloses can be concluded. Hemicelluloses are the most hydrophilic components of wood, thus, their decomposition also slightly weakened the water-bonding capacity of wood.

Future researches will focus on the improvement of polymerization, on PLA treatment of longitudinally compressed beech wood and on further monitoring of the polymerization products inside the wood.

## CONCLUSIONS

Lactic acid (LA) is an easily accessible substance that can be used to modify the properties of wood. We have information on the solid wood- LA relationship, but further experiments are needed. The aim of this publication is to examine the shrinkage and swelling of untreated and LA-treated beech samples, and also to determine the changes in the chemical composition of wood as a result of the treatment

Dry beech samples were impregnated with prepared LA oligomers under hot vacuum conditions. The impregnated samples were heated for 6 hours to polymerize the LA inside the wood. After sample preparation we performed high-performance liquid chromatography. As a result of the heating during the treatment, a small decomposition of hemicelluloses generated furfurals. The acetic acid content did not change significantly, while 12% of the LA in the samples remained monomeric, so the treatment has to be improved. During soaking of the samples, the loss of LA resulted in a weight loss of 31.4%. Thus, a lot of LA was leached and the shrinkage of the samples improved by only 36% as a result. It should be noted, however, that after the LA treatment, the water uptake and therefore the swelling of the absolutely dry samples were minimal, which is an outstanding result.

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## Improving effect of hydrophobized silica nanoparticles on the dimensional stability and wettability of wood

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### ABSTRACT

The aim of the research was to improve the dimensional stability of wood through bulk hydrophobization, as a result of impregnation with fluorinated silica nanoparticles. Two different wood species, beech (*Fagus sylvatica* L.) and scots pine (*Pinus Sylvestris* L.) were investigated. According to scanning electron microscopy imaging, fluorinated silica nanoparticles covered cell-wall surfaces mostly evenly, but discontinuously and could be observed as small, rough depositions. The presence of these hydrophobic nanoparticles further increased the dimensional stabilisation effect of the treatment by increasing the roughness of the cell wall surfaces, that leads to higher hydrophobicity. The impregnation with fluorinated silica nanoparticles was successful, as swelling decreased by 49-64% beside a low weight percent gain (0,74-1,16%), depending on wood species and treatment type. Significantly higher contact angles were observed for the treated wood surfaces. The observed hydrophobization effect was one of the main reasons for the improved dimensional stabilization of the treatment.

### INTRODUCTION

The demand for modified wood material increased worldwide, due to the commercialisation of different wood modification processes in the last decades. Wood modification processes indicate continuously new challenges. The aim is to get better performance from the wood, resulting in improvements in dimensional stability, decay resistance, weathering resistance, etc. Researchers carried out many studies to improve wood properties. One of the main conclusions of these studies was that the reduction of moisture adsorption is a key factor to improve durability or solve the problem of dimensional stability (Rassam *et al.* 2012; Sahin and Mantanis 2011).

A novel method to decrease water adsorption is the preparation of hydrophobic surfaces. To prepare low-surface-energy materials, one-step methods, like laser/plasma/chemical etching or lithography, and electrospinning are developed (Gonçalves *et al.* 2008; Kang *et al.* 2010; Han and Steckl, 2009). The hydrophobic surface can be fabricated through two-step processes as well, where the first step is the preparation of a rough film on the substrate, followed by a modification step to reach hydrophobicity (Saleema *et al.* 2011; Liang *et al.* 2014; Paul *et al.* 2011). As inorganic nanoparticles, SiO<sub>2</sub> is often used to fabricate nano-level roughness on surfaces due to its low cost and nontoxicity, followed by a subsequent modification to reach superb water repellency (Athauda *et al.* 2012; Zhou *et al.* 2013). Nowadays, superhydrophobic surfaces are a popular topic in various fields, because of their superb characteristics, such as low water or/and oil adhesion or self-cleaning (Sarkar and Saleema 2010; Guo *et al.* 2014). Studies on wood modification using “nano-silica” coating techniques on the cell walls are well known (Wang *et al.* 2013; Ebrahimi *et al.* 2017).

In this work, a simple method for hydrophobization of the cellular structure of wood, using fluorinated silica nanoparticles is shown. Silica nanoparticles are modified by a fluoroalkylsilane. Fluorinated nanoparticles are a good solution for preparing materials with low surface energy, to achieve hydrophobicity. This study aims to develop a simple method that can improve dimensional stability of the

treated wood material. The motivation for this work was to improve dimensional stability, by incorporation of functional materials such as fluorinated silica nanoparticles (FSNPs) not only as a surface treatment, but as a full-cross-section treatment. The goal of this study was to reach high dimensional stability using a low weight percent gain (WPG) treatment. The planned treatment will likely elongate the lifetime of the wood-based products, because the wood-water relations are essential at all utilization fields. The expected positive effect of the investigated treatment is the improvement of the dimensional stability of wood through a decreased moisture or water adsorption as a result of covering the cell wall surfaces by hydrophobic silica nanoparticles.

## EXPERIMENTAL

Wood samples of beech (*Fagus sylvatica* L.) and scots pine sapwood (*Pinus Sylvestris* L.) were cut into blocks of 20 mm × 20 mm × 30 mm (radial × tangential × longitudinal) and 10 mm × 50 mm × 50 mm (radial/tangential × tangential/radial × longitudinal). Silica nanopowder (15nm, 99,5%), 1H,1H,2H,2H-perfluorodecyltriethoxysilane (FAS-17, 97%), ethanol (ET, 99,99%) and distilled water was used for the preparation of the treatment.

The silica nanoparticles were further functionalized in an ethanolic fluoroalkylsilane solution. 100 ml ethanol and 1 g of silica nano powder were mixed by continuous stirring at 1000 RPM using a magnetic stirrer for 120 minutes at 50°C to form a homogeneous solution. FAS-17 was then added into this mixture while continuous stirring. 0,5 g or 1 g of FAS-17 was used to get two different level of fluorination. This step was resulting in the grafting of long perfluoro chains onto the surface of the nanoparticles to make them hydrophobic.

Wood blocks were vacuum impregnated with the prepared silica nano suspension. Wood blocks were oven dried at 105 °C in drying chamber before the impregnation process for 24 hours. The next step of the treatment was a 1 h long vacuum phase under 100 mbar pressure in a vacuum dryer at 25°C. This was followed by a 2 h long impregnation step under atmospheric pressure, by leaving the samples in the treatment suspension. The impregnated specimens were then placed in an oven at 60°C for 24 h, and at 105°C for another 24 h to let the ethanol evaporate.

To determine WPG, samples were weighed before the impregnation ( $m_0$ ), and after the curing step ( $m_{0, imp}$ ). WPG was calculated according to Eqn. (1):

$$WPG = \frac{m_{0, imp} - m_0}{m_0} [\%] \quad (1)$$

After treatment, the samples were investigated by scanning electron microscope (SEM). The surface was cut with a razor blade and coated with a sputter-coater before imaging. A Hitachi S-3400N type equipment, using an accelerating voltage of 10 kV and a working distance of 5 mm, was used for SEM imaging. 30 mm long and 20 mm wide samples were used.

To determine swelling for the calculation of anti-swelling-efficiency (ASE), 20×20×30 mm (radial × tangential × longitudinal) samples were used. There were 20 pieces for both wood species and treatment types used. 20 pieces of untreated samples for both wood species served as the control. The samples were dried at 105°C until a constant mass and then the dimensions were measured. Thereafter, the samples were submerged into water for 10 days and finally the dimensions were measured again. ASE was determined using radial or tangential swelling of untreated ( $S_{U,r,t}$ ) and treated ( $S_{T,r,t}$ ) samples according to Eqn. (2):

$$ASE_{r,t} = \frac{S_{U,r,t} - S_{T,r,t}}{S_{U,r,t}} \cdot 100 [\%] \quad (2)$$

To examine water repellency, contact angle (CA) of deionized water (surface tension: 3,2 mN/m) was evaluated as the ability of the resulting wooden surfaces to repel water. An optical goniometer (68-76 PocketGoniometer PGX+) was used to measure the CA of droplets on the prepared surfaces. Each droplet was dropped to the sample surface by vibrating the syringe. The volume of the droplet was controlled at around 4µL. CA was measured at intervals of 120 ms for the 1st s and at 5, 10, 20, 30, 60, 120, 240, 360, 480 and 570 sec. 20 CA determinations were made at different locations on the surface for each specimen.

## RESULTS AND DISCUSSION

### *Weight percent gain*

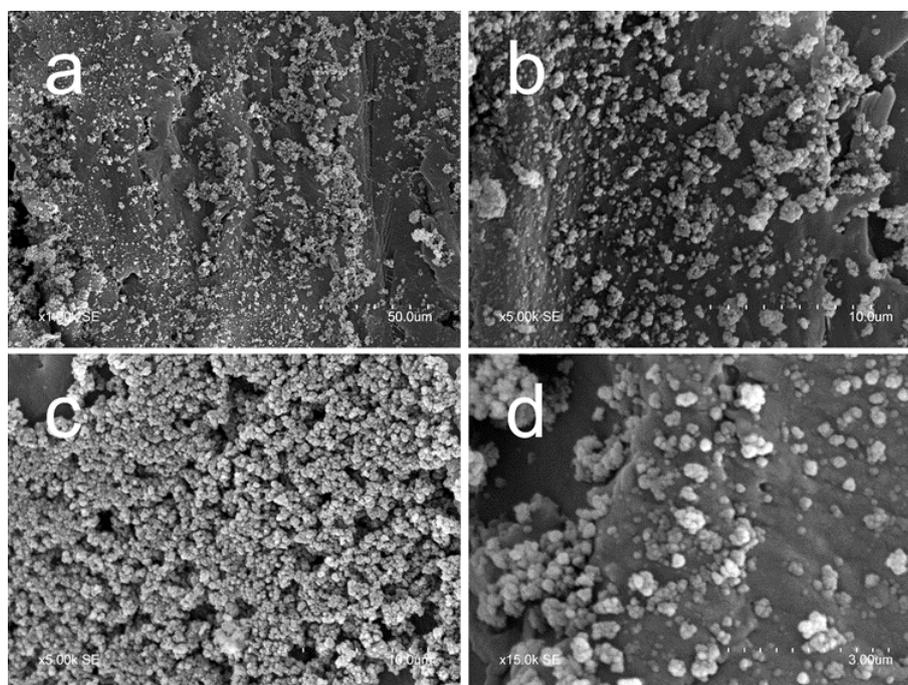
WPG values for tested beech and pine specimens are shown in Table 1. There were no significant differences in WPG of the different wood species. However, variation coefficients of beech samples' WPGs were significantly lower. This result shows a more even impregnation of beech material. The WPG reached with this treatment was in average 0,74-1,16%, depending on wood species and treatment type. This value is extremely low, compared to some other silica-based modification methods, where usually 20-60% WPG is reported (Donath et al. 2004).

**Table 1. WPG values of beech and pine samples as a result of FSNP treatment**

	Beech		Pine	
	SNP + 0,5% FAS-17	SNP + 1% FAS-17	SNP + 0,5% FAS-17	SNP + 1% FAS-17
	WPGdry [%]	WPGdry [%]	WPGdry [%]	WPGdry [%]
Mean	0,78	1,16	0,74	1,10
St. Dev.	0,06	0,07	0,10	0,17
Var. Coeff.	7,26%	5,83%	13,87%	15,77%

### *SEM imaging*

Presence of the FSNPs on the cell wall surfaces was stated by SEM imaging. FSNPs were distributed on the cell wall surface mostly evenly and could be observed as small, rough depositions (Figure 1 a, b and d). The FSNPs on the cell wall surfaces showed also some larger deposits, agglomerations in the cell lumens of fibres, tracheids and rays (Figure 1 c). However, these depositions did not fill the entire lumen, they might further increase the dimensional stabilisation effect of the treatment. These results show that the treatment does not produce a continuous coating, but it increases the roughness of the cell wall surfaces, that leads to higher hydrophobicity. The good penetration, the effective size and uniform distribution of nanoparticles may reduce the hygroscopicity and improve the dimensional stability of wood as well.



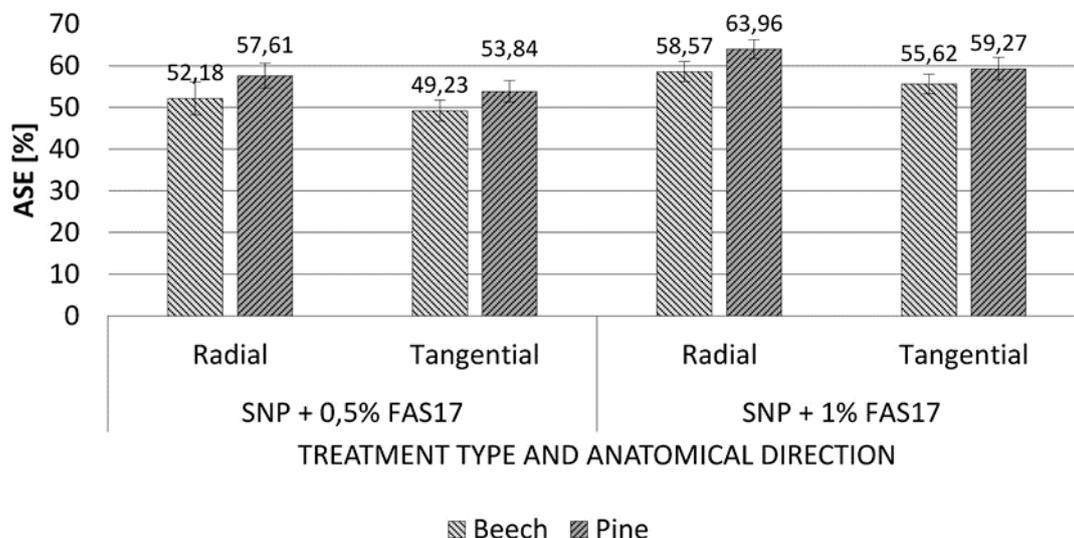
**Figure 1. SEM images of the treated wood material showing FSNPs on the cell wall surface (a, b and d), and the structure of agglomerations on the cell wall (c)**

The reason for covering the cell walls discontinuously by FSNPs is the low concentration of the FSNP suspension (1,5 and 2 wt%). This fits with the goal to keep the amount of FSNPs low in the wood. The loose/porous structure of the FSNP deposits is well illustrated in Figure 1 c. FSNPs are spherical blocks, but the determination of the diameters is difficult, because they overlap in the image. A rough estimation gives a globule size between 50 and 200 nm.

### **Anti-Swelling-Efficiency (ASE)**

Shrinking and swelling properties decreased remarkably in case of both investigated wood species and treatments (Figure 2). FSNP treatment resulted in slightly, but significantly lower ASE in both radial and tangential direction in case of beech wood, compared to pine. These results show that, however not much, but wood species has some effect on the efficiency of the investigated treatments. This is related to the differences in the anatomical structure that leads to different permeability of beech and pine in the different anatomical directions. FSNPs covered the cell wall surface, that delays and prevents the penetration of water molecules to the cell wall. The higher ratio of FAS17 resulted in slightly higher ASE values. This indicates that fluorination of the silica NPs was more effective using higher amount of FAS17 in the treatment solution. Despite, that slightly higher WPG values for beech were observed (Table 1.), this difference was not realized in the ASE of the treatments, as no correlation was found between the WPG and ASE of the treated beech and pine samples in radial and tangential direction.

ASE was found high, despite of the low amount of FSNP in wood used as modifying agent. The improvement in dimensional stability can be attributed to the void spaces being occupied by nanoparticles (Devi and Maji 2012) and by the reduced hygroscopicity of the treated wood material (Soltani *et al.* 2013; Habibzade *et al.* 2016; Kumar *et al.* 2016). Other authors also assume, that the nano-based compound treatment acts on the wood-water interaction possibly with its molecules reducing the surface energy, thus, supporting the dimensional stability with water (Sahin and Mantanis 2011). Thus, it seems that accessibility of sites that enable bonding of water molecules is reduced by FSNP layers on the cell-wall surfaces.



**Figure 2.** ASE as a result of FSNP treatments on beech and scots pine wood in different anatomical directions (whiskers showing standard deviation of the results).

### **Water repellency**

Results showed high hydrophobization effect as a result of the investigated silica-based treatment (Figure 3). The initial CA (at time = 0 s) of the untreated wood material was between 65-70° for both wood species. In contrast to that, significantly higher CAs were observed for the FSNP treated wood surfaces (between 130-145°), depending on the treatment type. Higher ratio of hydrophobic modifier resulted in improved water repellency, as the addition of 1% FAS17 increased the CA significantly, by additional 15° in average.

There was no difference observed between wood species regarding CA. This is close to the superhydrophobic region ( $>150^\circ$ ). The treatments are not only providing high hydrophobicity for wood, but they also provide long lasting effect, as the contact angle is only moderately decreasing with time. Water droplets rested steadily on the hydrophobic wood surface, which is in contrast to the instant penetration of water into the pristine wood.

Thus, the fluorinated SNP treatment is a stable treatment. These results are supporting the conclusions taken related to the dimensional stabilization and water-uptake decreasing effect of the investigated treatments. One of the main reasons, why these treatments are improving these properties of wood is the long-lasting hydrophobization effect of the treatments. Improving effect of nanoSiO<sub>2</sub> on water repellency have been also reported by other researches. Hosseini *et al.* (2014) created fiber reinforced composites based on lignocellulosic waste material in combination with nano-SiO<sub>2</sub>. The composites showed a useful water repellent property as a result of the nanoparticles. Other study also demonstrated the efficiency of perfluoroalkyl methacrylic copolymer and silica nanospheres based fluoro-containing silica nanocoating on wooden substrates resulting in superhydrophobicity (Hsieh *et al.* 2011).

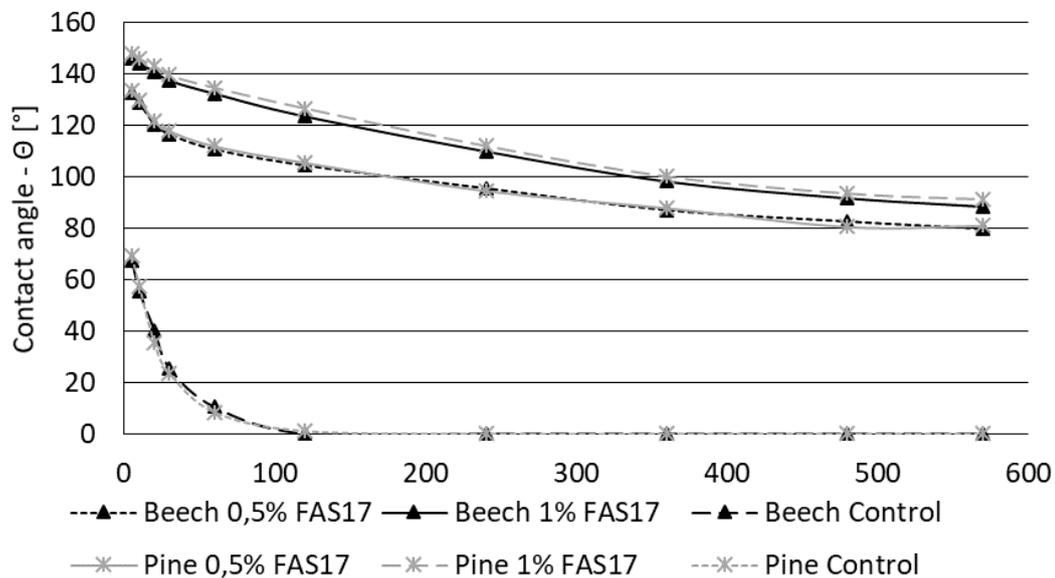


Figure 10. Effect of silica FSNP treatments on the contact angle of beech and scots pine wood.

## CONCLUSIONS

It was proved that it is possible to improve dimensional stability of wood with the use of fluorinated silica nanoparticles. Swelling decreased remarkably, where the ratio was depending on wood species and anatomical direction. The ASE was similar in radial and tangential direction for both beech and pine; however, a slight, statistically significant difference could be observed in the results between the different wood species. The improved hydrophobicity of the cell wall surfaces through the deposition of fluorinated silica nanoparticles make the investigated treatments more effective against liquid water, compared to water vapour. SEM imaging showed that the distribution of the nanoparticles is mostly even on the cell wall surfaces, but some small, rough depositions were found as well. The nanoparticles act as a coating on the cell wall surfaces.

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## Response of wood anatomical traits of *Quercus ilex* to drought stress: a case study from a plantation in Southern Italy

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**Keywords:** wood, anatomy, holm oak, drought stress, plantation, Mediterranean area.

### ABSTRACT

*Quercus ilex* (holm oak) is one of the most widespread evergreen oaks in the Mediterranean basin, growing from the sea level up to about 600 m asl. *Q.ilex* stands are complex ecosystems from which can benefit a whole city or community. The wood of *Q.ilex* is difficult to work with, but it can be finished very well. In recent years, various natural disturbances enhance the vulnerability of Mediterranean urban forests and thus modify their function.

In this context, our aim was to analyse the characteristics of the wood of *Q.ilex*, growing in a Mediterranean plantation in Southern Italy, during the particularly dry year 2017.

To pursue our aim, we collected microcores containing xylem samples at biweekly intervals from 8 dominant trees during the year 2017. Thin cross sections were cut from microcores with a rotary microtome and analysed by means of a light microscope.

Our results showed that the wood formed during the considered year was characterized by features indicating the occurrence of a stress event. In particular we found the occurrence of intra-annual density fluctuations, discontinuous rings along the circumference of the sections (wedging rings) and tyloses.

In conclusion, the features of *Q.ilex* indicate that the trees were probably suffering stress due to the drought conditions occurring during the year 2017. This is a valuable information to plan a preservation action through a proper management.

### INTRODUCTION

*Quercus ilex* (holm oak) is one of the most widespread evergreen oaks in the Mediterranean basin, growing from the sea level up to about 600 m asl. In Italy, *Q.ilex* is also commonly planted for ornamental and urban green purposes. *Q.ilex* stands are complex ecosystems from which can benefit a whole city or community. Their benefits include a high ecological, recreational and landscape value and supply. The wood of *Q.ilex* is difficult to work with, but it can be finished very well. It is resistant to pests. It is used mostly as fire wood and for obtaining charcoal, but also for coarse carpentry (handles, pieces for presses, levers, pins, wagon axles, lists for the transport of stones, railway sleepers, beams, furniture, parquet).

In recent years, various natural disturbances such as droughts, heatwaves, and the increasing amounts of pests, likely due to climate change, affect the wood formation and structure and, consequently, enhance the vulnerability of Mediterranean urban forests and thus modify their function (Steenberg et al. 2017). Survival of forest tree species in semi-arid environments such as the Mediterranean area is threatened by the increase in harsh drought conditions.

*Q. ilex* is more vulnerable to xylem embolism than are other Mediterranean species (Martinez-Vilalta et al. 2002) for this, is likely to be replaced in the future by oak species resistant to low water availability (Liu et al. 2018).

In this context, our aim was to analyse the characteristics of the wood of *Q. ilex*, growing in a Mediterranean plantation in Southern Italy, during the particularly dry year 2017. Our study aims to provide further knowledge of wood formation process of Mediterranean species to develop strategies for future sustainable management.

## EXPERIMENTAL METHODS

### *Study site*

The selected study area is “Parco Gussone” (40° 48’ 40.3’’ N, 14° 20’ 33.8’’ E) (Fig.1), the former park residence of the Bourbon Kings of Naples where *Q.ilex* was planted in 1738 for landscape and hunting reasons. Here already grown trees were implanted on a lava flow of 1631 detonated with dynamite. Today, the park consists in an old grown dominant evergreen *Q.ilex* trees with the occurrence of other deciduous species such as *Fraxinus ornus*, *Quercus pubescens*, *Ulmus campestris*, and *Celtis australis*.



Figure 1: Sampling site in “Parco Gussone” (40° 48’ 40.3’’ N, 14° 20’ 33.8’’ E), Naples.

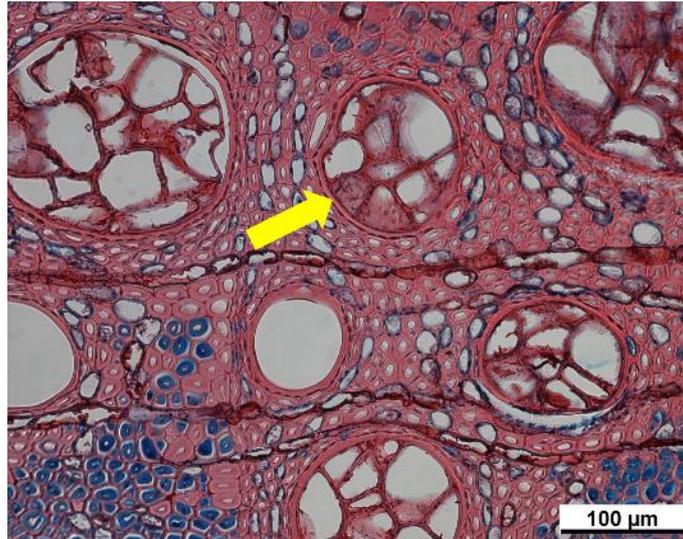
### *Anatomical analysis*

We collected microcores containing xylem samples at biweekly intervals from 8 dominant trees during the year 2017.

Sampled microcores were stored in FAA (formalin, acetic acid and ethanol), and after a week, dehydrated ethanol series, cleaned with D-li-monene and embedded in paraffin blocks (tissue processor Leica TP1020-1, Nussloch, Germany). Thin cross sections (9 µm thick) were cut from microcores with a rotary microtome (RM 2245 Leica, Nussloch) subsequently stained with a safranin and astra blue water solution. Obtained cross sections were observed under a Nikon Eclipse 800 light microscope we observed the slides and microphotographs were taken by a digital camera (DS-Fi1) connected with the NIS-Elements BR 3 image Analysis System (Melville, NY, USA). Wood increments and its characteristics were analysed during the period of formation during the year 2017.

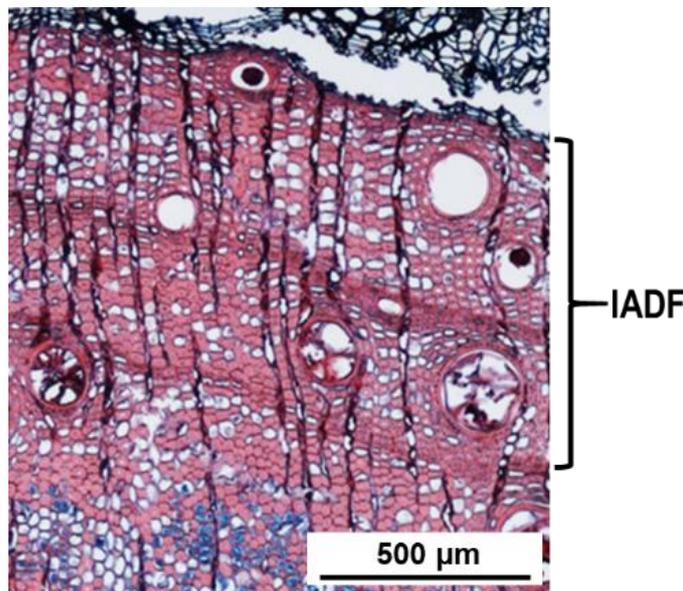
## RESULTS AND DISCUSSION

Our results showed that the wood formed during the considered year was characterized by features indicating the occurrence of a stress event. Signal of drought stress suffered by the trees during their growth can be recorded in the wood structure. Indeed, some wood traits, such as the size of conductive elements and cells wall thickness, are proportionally related to the efficiency and safety of water transport (De Micco and Aronne 2007). Under stressful conditions, primed by either biotic or abiotic factors, embolism can occur and there happens a compartmentalization process culminating in vessel occlusion by tyloses or gums, depending on the size of conduits (De Micco et al. 2016a). In particular we found the occurrence of tyloses (Fig.2), Intra-annual density fluctuations (IADFs), discontinuous rings along the circumference of the sections (wedging rings).



*Figure 2: Tyloses (yellow arrow) occurring in the vessel lumen of Q.ilex.*

We detected and classified IADFs (Fig.3) in accordance with De Micco et al. (2016b). IADFs are regions within a tree ring where abrupt changes in density occur (De Micco et al. 2016b) that can be formed within the tree ring in response to environmental changes during the calendar year that affect cambial activity and cell differentiation. In this case, they were formed as result of stress condition during the summer periods.



*Figure 3: Intra-Annual Density Fluctuation (IADF) formed during the year 2017.*

## CONCLUSIONS

In conclusion, the features of *Q.ilex* wood formed during the year 2017 indicate that the trees were probably suffering stress due to the drought conditions in summer. The analysis of IADFs and occurrence of tyloses may provide accurate information at the intra-annual level, allowing a deeper understanding of climate-growth relationship. This kind of information are valuable to plan a preservation action through a proper management.

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## Effect of moisture content on the wood surface roughness measured on birch and black locust wood surfaces

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**Keywords:** new method, wood moisture content, birch, black locust, distilled water, diiodomethane, wood surface roughness.

### ABSTRACT

Test results can be compared only if the test conditions are similar. Moisture content (MC) of wood influences adhesion of different lacquers and adhesives, whilst wood surface roughness is also in relation with the moisture content of the wood. Contact angle, wood moisture content and wood surface roughness are 3 interdependent parameters. The problem arose that with increasing moisture content of wood samples the surface roughness also varies. In this study we focused on finding a test procedure which supports minimizing the distortive effect of varying surface roughness during a swelling or shrinkage process, when for example adhesion is aimed to be measured. The investigated procedure was: first kiln drying the samples to a moisture content around 6%, than soaking the samples into water till they reach a moisture content higher than 30%, than performing contact angle measurements on differently moist surfaces during a drying process. Wood MC and surface roughness was measured on birch (*Betula pendula*) and black locust (*Robinia pseudoacacia*) surfaces. Between the MC of wood and wood surface roughness of birch, a linear relation  $y=ax+b$  (with high correlation) was found. In case of wood species with evenly distributed, small pores,- like birch,- it is expected, that the equation has the same form, the difference is expected to arise in the constants of the equation. It is very likely that the different wood species have their own equation in the upper context. Study will follow on a wider range of wood species. The evaluation of the large-porous species, like black locust, needs further effort. Filtering the data, which belong to the large, open pores, may be a solution to enable the evaluation of changes occurring in the ground tissue, as result of MC changes.

### INTRODUCTION

Surface quality of machined wood products is of major importance both during the finishing process and during service. There are several studies reporting on measuring and evaluation of wood surface roughness of different wood species and wood products, but standards prescribing the test conditions in case of wood are still missing. There aren't recommendations regarding the convenient moisture content of wood in standards. Wood surface roughness is one of the main parameters of surface quality evaluation. Can be measured in multiple ways and there are several standardised and non-standardised parameters of evaluation. Average roughness ( $R_a$ ), mean peak-to-valley height ( $R_z$ ) and maximum roughness are some of the parameters that are used in order to characterize wood surface (Kilic et al. 2006).

Roughness status of wood has many influencing factors like the anatomical structure of wood, density, moisture content, the type of machining tool and its speed (Aguilera and Munoz, Gurau et al 2005, Magoss 2008). Korkut and Guller (2007) investigated the effects of heat treatment on surface roughness of red-bud maple (*Acer trautvetteri* Medw) at 12% MC. Results indicated that surface roughness decreased with increasing temperature treatment and treatment times. Vitosyte et al. (2012) studied the effect of surface roughness on adhesion strength of tangential planed defect free kiln-dried ash (*Fraxinus excelsior* L.) and birch (*Betula* L.) samples at  $10\pm 1\%$ .

The aim of the present study was to evaluate the effect of MC on the surface roughness, measured on birch (*Betula pendula*) and black locust (*Robinia pseudoacacia*) surfaces, conditioned to different moisture

contents from 6% MC up to 30% MC, in order to find a test procedure which minimizes the distortive effect of varying surface roughness during a swelling or shrinkage process, when for example adhesion is targeted to be measured.

## MATERIALS AND METHODS

In this study, samples from birch (*Betula pendula*) wood and black locust (*Robinia pseudoacacia*) with dimension of 50 mm x 80 mm x 20 mm were prepared. The surfaces were machined with sand paper of grit size 120, because sanding results a relatively uniform, smooth wood surface (Magoss, 2005; Brahmia et al. 2020 ;Gurau et al.2009). Samples were immersed into distilled water until reaching a moisture content above 30%. After soaking, all the samples were placed into a dryer, to  $60^{\circ}\text{C} \pm 3$ . The MC of the samples started to decrease. A temperature-MC diagram was used, to set the drying time and to estimate the expected MC of the samples; however the actual MC of the samples was measured by The Kern MLB 50-3N Moisture analyzer instrument. This instrument was made for fast and reliable determination of material MC by applying thermogravimetric method. Thin slices from the sample's sides were cut. The slices were placed on the sample holder plate of the Kern MLB 50-3N Moisture analyzer instrument, which displayed digitally the actual MC value after a few minutes. Ten roughness measurements were performed on 5 samples, in each moisture content range. On each tested sample, surface roughness was performed perpendicular to the grain, using MAHR S2P Perthometer instrument, equipped with a stylus tip of 2  $\mu\text{m}$  radius, providing roughness data along the measuring trace. The unfiltered P,- primary profile was evaluated by  $R_a$  mean roughness,  $R_z$  the average maximum peak to valley height and  $R_{\text{max}}$  maximum roughness (Magoss 2008), as per DIN EN ISO 4288 standard. The samples were tested during a drying process, similar to the natural process of wood drying, at the following moisture contents: 29.97%, 17.96%, 16.20%, 14.11%, 12.16%, 10.10%, 7.99% and 6.03. The average MC values of the samples will be referenced in the followings into the text as rounded to the closest integer value.

## RESULTS AND DISSCUSION

Based on the results can be stated that birch as wood species was suitable for the evaluation of a convenient test procedure, as its anatomy enables the identification of changes in surface roughness, occurring during a shrinking/swelling process. Birch was a good choice as contains evenly distributed pores and has relatively uniform tissue structure. Black locust contains large open pores which influence the roughness values but don't contribute to the changes occurring in the ground tissue of the surface. They distort the measured roughness of the surface proportionate to the number of large open pores along the measuring trace. The more deep open pores are measured along the measuring trace the more irrelevant become the roughness changes of the ground tissue. Table 1 represents the average  $R_a$  and  $R_z$  values of birch and black locust samples. Neither the average roughness  $R_a$  values, neither the average peak to valley height  $R_z$  values of black locust don't follow any well-defined trend or function. The evaluation of roughness of the large-porous species, like black locust, needs further effort. Filtering the data, which belong to the large, open pores may be a solution to enable the evaluation of changes occurring in the ground tissue, as result of MC changes.

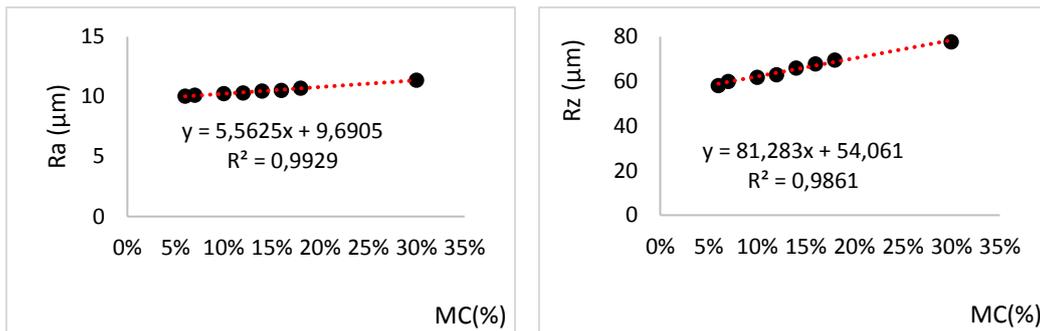
Birch displays different data. Both the  $R_a$  and  $R_z$  roughness values of birch follow a linear trend with increasing moisture content of the samples. Between 6 % and 18% MC there isn't a significant difference between the  $R_a$  values of birch samples. The linear equation is mostly horizontal, showing, that by the suggested test procedure it is possible to decrease the roughness changes of the surface in the region of 6%-18% MC. Around 30% MC the surface roughness is high and increases the slope of the function and results linear equation of the form  $y=5,56x+9.69$ . In that region of moisture contents which are frequently used/reported in experiments because are typical to artificially dried samples (6%-18% MC) the  $R_a$  roughness values of birch don't differ significantly, their linear equation is mostly horizontal. The straight line gets its slope due to the roughness values around 30% MC.

The function of  $R_z$  values in mirror of MC is also linear. The moisture content of samples around 30% results high roughness values of  $R_z$ . There aren't significant differences between two consecutive  $R_z$  values from 6% and 18% MC (when  $p=0,05$ ,  $t_{6-8}=0,678131$  ,  $t_{8-10}=0,652915$  ,  $t_{10-12}=0,751699$  ,  $t_{12-14}=0,400554$  ,  $t_{14-$

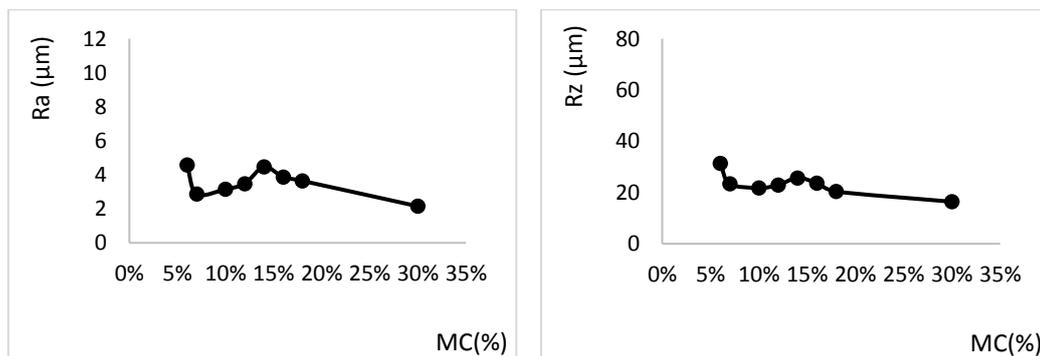
$t_{16}=0,574530$  ,  $t_{16-18}=0,552955$  ). In the same time  $R_z$  is a more sensitive parameter than  $R_a$  and if we compare the  $R_z$  values of 6%MC with the  $R_z$  values belonging to 16%, 18% and 30% MC, they are significantly different ( $t_{6-16}=0,014581$  ,  $t_{6-18}=0,011967$  ,  $t_{6-30}=0,000062$ ). Around 16% and 18% MC the  $R_z$  values are somewhat increased relative to the one of 6% MC. This shows, that the slope of the straight line alters from the horizontal in that measure, that for 6% to 14% MC it can be expected that  $R_z$  roughness values stay significantly unchanged, whilst from 16% MC up, the roughness has influence on the measured data. Based on  $R_a$  data the whole MC range from 6% to 18% has uniform roughness, the same evaluation based on  $R_z$  values supports the upper statement between 6% and 14% MC. In this MC region roughness doesn't distort the adhesion, contact angle etc measurements values. The preconditioning of samples according to the procedure recommended in this paper, may be suitable for the elimination or at least reduction of the changes in surface roughness during a drying process.

**Table 1:  $R_a$  average roughness ( $\mu\text{m}$ ),  $R_z$  the average maximum peak to valley height ( $\mu\text{m}$ ) and  $R_{\text{max}}$  maximum roughness ( $\mu\text{m}$ ) of birch and black locust samples registered during drying at different levels of MC (%)**

		6.03%	7.99%	10.1%	12.16%	14.11%	16.2%	17.96%	29.97%
Birch	$R_a$ ( $\mu\text{m}$ )	10.40	10.12	10.25	10.31	10.47	10.52	10.72	11.38
	$R_z$ ( $\mu\text{m}$ )	58.182	60.03	61.91	63.03	65.96	67.72	69.63	77.68
	$R_{\text{max}}$ ( $\mu\text{m}$ )	81.88	83.67	85.22	87.91	90.55	93.54	95.02	108.08
Black locust	$R_a$ ( $\mu\text{m}$ )	4.581	2.879	3.148	3.481	4.474	3.859	3.642	2.157
	$R_z$ ( $\mu\text{m}$ )	31.4	23.4	21.8	22.9	25.7	23.7	20.4	16.4
	$R_{\text{max}}$ ( $\mu\text{m}$ )	44.4	48.5	34.6	32.8	30.8	28.7	24.1	20.7



**Figure 1:  $R_a$  and  $R_z$  roughness values ( $\mu\text{m}$ ) of birch samples registered during drying**



**Figure 2:  $R_a$  and  $R_z$  roughness values ( $\mu\text{m}$ ) of black locust samples registered during drying**

## CONCLUSIONS

Birch as wood species was found suitable for the evaluation of a convenient preconditioning procedure as its anatomy enables the identification of changes in surface roughness, occurring during a shrinking/swelling process, because contains evenly distributed pores and has relatively uniform tissue structure. Black locust contains large open pores which influence the roughness values but don't contribute to the changes occurring in the ground tissue of the surface. They distort the measured roughness of the surface proportionate to the number of large open pores along the measuring trace. The recommended sample preconditioning procedure was found suitable for to reduce the changes of surface roughness occurring during a drying process.

## ACKNOWLEDGEMENT

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## **Changing climate and adaptive forest management: when today's „best practices” are no longer sufficient**

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### **ADAPTATION AND DECISION SUPPORT IN FORESTRY**

„We are shooting a moving target” when we talk about the effects of climate change, therefore timely solutions are necessary for constantly changing conditions. It cannot be forgotten that in solving the problems of climate change affecting forestry, there is only one key player apart from intelligent people, the living plant itself. We must recognize the adaptation processes of our species and introduce new management practices in line with the speed of the human-induced climate change. Researchers are responsible for collecting and evaluating available information. All these data, which had been accessible separately for a long time, had to be made easily available for some sites of actual intervention using only the toolkit provided by GIS and applying new evaluation methods.

Accepting the month average rainfall and temperate data of given sites by climatologists, the future climatic conditions can be determined, using the newly developed drought indicator (FAI). So it is a realistic idea that future sites condition are predictable and, therefore, we can make suggestions about the target stocks, tree species and even the origins and sources of propagation materials from tree species safe to apply for the next decades.

### **TODAY'S "BEST PRACTICES" MAY NOT BE ENOUGH WHEN WE TALK ABOUT CLIMATE CHANGE EFFECTS ON FORESTRY**

#### ***Site as a time-varying trait***

Declining precipitation and above the average warming up is forecasted in central-eastern Europe. A specialty of this area is that most dominant species reach here the xeric limit of their distribution. Consequently, the majority of the native species are directly threatened by progressive warming and increasing drought frequency.

#### ***How will trees respond within a generation?***

The change is 10 to 100 times faster than what the tree species were able to follow in their evolution. Impacts already visible:

- fire frequency
- abiotic calamities
- pests and diseases
- production decline
- mass mortality effects
- change of distribution.

#### ***Climate stress is an effective selection agent on genetic resources***

The loss of genetic diversity decreases the adaptive potential possible consequences:

- mass mortality effects
- loss of habitat
- changes of tree species distribution.

### ***Active human assistance needed***

Instead of native species introduced drought-tolerant exotic species should be planted immediately? It's good news that there are still enormous scope for indigenous species, which we have largely left untapped! Solution: to determine drought tolerant populations within native species and to use their propagation material as supplementary seed source in natural regeneration.

### ***Forecasts***

- Prediction the new site type variants
- Selection of species to the future sites
- Selection appropriate propagation material within species.

Site consists largely stable factors as a soil type, depth, texture, hydrology but climate has shown fast change in the last decades. New drought indicator (Forest Aridity Index - FAI) (Eq. 1) has been developed that characterizes the climate categories with meteorological variables and therefore expected changed can be forecasted:

$$FAI = 100 \cdot T_{VII-VIII} / (P_{V-VII} + P_{VII-VIII}) \quad (1)$$

## **KNOWLEDGE OF THE ORIGIN OF PROPAGATION MATERIAL IS NECESSITY AND NOT LUXURY - ASSISTED SEEDLOT TRANSFER IS A SOLUTION OF RAPID ADAPTATION OF NATIVE FOREST**

### ***Proactive seedlot selection support***

Solving the problems caused by climate change is the well-chosen plant itself. Forest reproductive material zones are based on geographic constraints (ecology is only indirect) today. It is suggested to keep the forest reproductive materials within the zone of origin. New approach needed: classification of reproductive material zones are based exclusively on ecological parameters without geographic constraints. Online service helps to find seed-sources with projected condition (2050) for the target species. Moving of the seedlots is aimed to introduce pre-adapted populations to get prepared for the situations expected in 2050!

### ***The future already has begun***

Currently available support for human-assisted seed transfer. Improve public awareness of ecological and evolutionary processes, instead of intuitive and emotional-guided misinterpretations. Ultimately the rapid adaptation of forests depends on prudent use of reproductive material.

## Selected physical and mechanical properties of beech wood treated by caffeine

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**Keywords:** caffeine, wood modification, beech, density, compression strength, hygroscopicity.

### ABSTRACT

Beech is one of the most important species in the Central Europe. With decreasing share of spruce wood on the market its importance will be even growing and it can be expected to increase its use in the region. Beech wood has excellent mechanical properties but very low resistance against biotic degradation agents. Currently, its durability is increased by traditional fungicides. However, demands for more environmentally friendly procedures are increasing and new non-traditional procedures are being explored. One of the investigated possibilities is the use of caffeine, where a biocidal effect has been demonstrated in some works. In this study, the effect of beech wood modification by caffeine and its impact on some physical and mechanical properties was tested. The beech lumber was used to prepare the test specimens. Test specimens 20x20x30 mm (radial x tangential x longitudinal) were divided into 4 groups. The first group of test specimens was treated with 2% caffeine solution, the second group of samples was soaked in distilled water, the third group of test specimens was the reference and the last group of the testing samples was dipped in 2% caffeine solution and subsequently exposed to the accelerated weathering. The effect of the treatment on selected physical and mechanical properties, specifically wood density, swelling, compressive strength were tested. All the tests were performed according to national and European standards. No significant effect of caffeine modification of beech wood was confirmed in the case of the tested physical and mechanical properties.

### INTRODUCTION

Beech (*Fagus sylvatica*, L.) plays an important role not only in European forestry but also provide timber, which is widely used in industry. Its wood is quite hard and heavy, with high strength and stiffness. One of its disadvantages is high shrinkage (Wagenführ 2007). The biggest limitation, from a view of industry, is low durability and resistance against fungi (EN 350). One of the ways, how to increase the resistance of wood, is impregnation. Most of the stuffs, used for impregnation nowadays, are synthetic biocides (Reinprecht 2016). As an opinion of society has change in the course of time, the stuffs on natural origin are preferred for impregnation of wood. Several studies tested substances of natural origin, which are supposed to have low impact on environment, and their effect on wood resistance (Hill 2011, Hill and Norton 2014, Pánek and Reinprecht 2016). One of those natural stuffs is caffeine. Improvement of wood resistance against wood-destroying fungi and moulds was confirmed in some studies (Arora and Ohlan 1997, Ratajczak et al. 2018, Kwaśniewska-Sip et al. 2018, Kwaśniewska-Sip et al. 2019). Modification of wood, including impregnation, usually has more or less serious impact on wood structure. Substances, used for the impregnation, penetrate the cell wall, which is consequently reflected in wood properties (Borůvka et al. 2016, De Meijer 2005, Doubek et al. 2018, Hill 2006).

In this paper, we present the first results of the project focused on beech wood modification by caffeine in order to improve the wood resistance against fungi and moulds. As these tests are long lasting, the aim of this study is to provide the effect of such modification on some physical and mechanical properties that are important from a view of industry. We tested wood density, swelling and compression strength of beech wood modified by caffeine solution in contrast to unmodified wood. Moreover, we evaluated the impact of artificial accelerated weathering on those properties.

## EXPERIMENTAL METHODS

Central boards of beech wood (*Fagus sylvatica*, L.) were used to prepare the testing material. Laths, with dimensions 20 × 20 mm (radial × tangential) were cut from the beech lumber. Test specimens with dimensions 20 × 20 × 30 mm (radial × tangential × longitudinal) were subsequently cut from each lath, in the number of 4 pieces per lath. This number is intended to provide longitudinal parallelism for four series of experiments for each evaluated property. The testing specimens did not contained any knots, cracks and the allowed deflection of the fibres in the longitudinal direction was less than 5 °.

The first series of the testing samples served as a reference (without any modification), the second series of the testing samples was dipped in distilled water, the third series of the testing samples was dipped in 2% caffeine solution and the last series of the testing samples was dipped in 2% caffeine solution and subsequently exposed to the accelerated weathering. 30 testing samples for each series per the tested properties were used. After the modification, the samples were air-conditioned until the equilibrium moisture content was obtained. We used CLIMACELL 707 air-conditioning chamber (BMT Medical Technology Ltd., Brno, Czech Republic) with relative air humidity (65 ± 5)% and temperature (20 ± 2) °C. For artificial accelerated weathering, we used Xenotest Q-Sun Xe-3 (Q-Lab, Cleveland, OH, USA) with repeating steps 2.5 hour of irradiation exposure and 0.5 h of spray with distilled water. Temperature of air was 45 °C, temperature at black panel 80 °C, Air humidity φ = 30%, TUV = 55 W.m<sup>-2</sup>, Total energy during 1000 hours of test 167 530 kJ.m<sup>-2</sup>. All of the tests on the physical and mechanical properties presented in this paper were performed according to the Czech national standards. From the physical properties basic wood density, oven-dry density, volumetric swelling were tested and from the mechanical properties, the compression strength along fibres was evaluated.

### *Density*

Basic density was calculated as the proportion of wood dry mass and its volume in the swollen state. We used 30 testing samples per modification. The same testing samples were used to evaluate oven-dry density, which was calculated as a proportion of wood dry mass and its volume in oven-dry state. Laboratory kiln and conditions 103 ± 2 °C were used to obtain oven-dry state of wood. All those procedures followed the national standard (ČSN 49 0108, 1993) and we used these formulas (Eq. 1 and Eq. 2):

$$\rho_0 = m_0 / V_0 \quad (1)$$

Where  $\rho_K$  is the oven-dry density (kg.m<sup>-3</sup>),  $m_0$  is the weight of the oven-dry testing samples (kg),  $V$  is the volume of the oven-dry testing samples (m<sup>3</sup>).

$$\rho_K = m_0 / V_{\max} \quad (2)$$

Where  $\rho_K$  is the basic wood density (kg.m<sup>-3</sup>),  $m_0$  is the weight of the oven-dry testing samples (kg),  $V$  is the volume of the testing sample with the moisture content higher than the fibre saturation point (m<sup>3</sup>).

### *Swelling*

Dimensional changes of wood related to moisture content changes was tested as a difference between the volume of saturated testing sample and the volume of oven-dry sample related to the oven-dry volume. 30 testing samples per modification were used to evaluate volumetric swelling. Drying, as part of this test, was performed using the Binder FD 115 laboratory kiln (Binder Inc., Tuttlingen, Germany) at 103 ± 2 °C. The test was performed in accordance with the national standard (ČSN 49 0126, 1989) using the following formula (Eq. 3):

$$\alpha_v = (V_{\max} - V_{\min}) / V_{\min} \cdot 100 \quad (3)$$

Where  $\alpha_v$  is volumetric swelling (%),  $V_{\max}$  is the volume of the testing sample with the moisture content higher than the fibre saturation point,  $V_{\min}$  is the volume of the testing sample with moisture content 0% (oven-dry volume).

***Compression strength***

Compression strength parallel to the grains was determined using the Tira 50 kN testing machine. We used 30 testing samples of beech wood per modification. Before the test, the testing samples were exposed to conditions of  $20 \pm 2$  °C and  $65 \pm 5$  % air humidity to ensure for wood equilibrium moisture content of 12%. The national standard (ČSN 49 0110, 1980) and this formula were used (Eq. 4):

$$\sigma = F_{\max} / a.b \tag{4}$$

$\sigma$  is the compression strength along the fibres (MPa),  $F_{\max}$  is the maximal load (N), a.b is the cross section of the testing sample (mm<sup>2</sup>).

For the statistical analysis, analysis of variance ANOVA was used to evaluate the significance of individual factors. Duncan’s Multiple Range Test was used to compare the properties of wood among the different treatments. The same significance level of  $\alpha = 0.05$  was used for all of the analyses.

**RESULTS AND DISCUSSION**

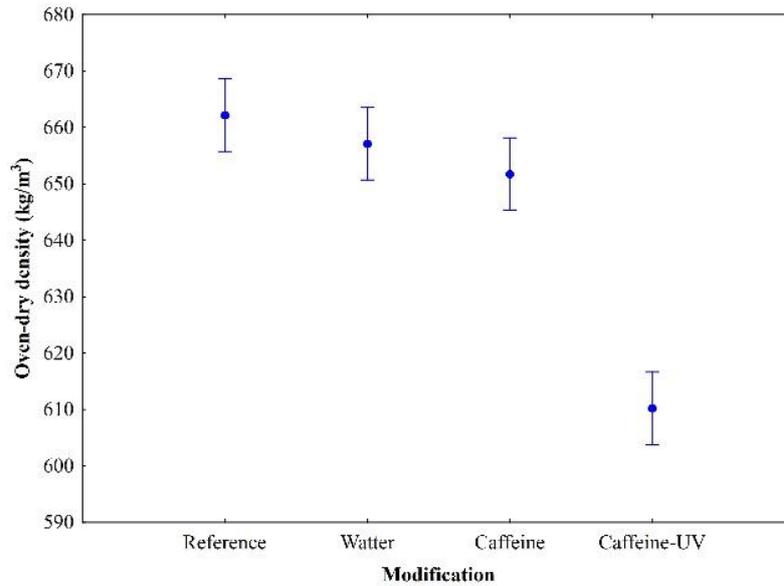
Table 1 shows results for the tested physical and mechanical properties. It is obvious from the tab that effect of modification is not striking from a view of applications. Such wood could be using in industry for different purposes without serious impact on the properties. Only exception is the wood modified by caffeine and subsequently exposed to UV radiation, where evident decrease in values (especially in the case fo the physical properties) was recorded. If we applied statistical analysis, it is evident from the graphic outputs (from Fig. 1 to Fig. 4) that that there is certain statistically significant negative effect of caffeine, both on the basic physical quantity, it means density, and logically on the compression strength along the fibres.

*Table 1: Tested physical and mechanical properties - mean (standard deviation)*

Modification	Oven-dry density [kg/m <sup>3</sup> ]	Basic density [kg/m <sup>3</sup> ]	Volumetric swelling [%]	Compression strength [MPa]
Reference	662 (12)	691 (14)	23.3 (0.8)	58.1 (1.9)
Water	657 (9)	686 (13)	22.4 (1.1)	58.3 (1.6)
Caffeine	652 (8)	676 (20)	21.4 (0.5)	55.9 (3.5)
Caffeine-UV	610 (9)	655 (18)	17,2 (1,1)	52,9 (4,4)

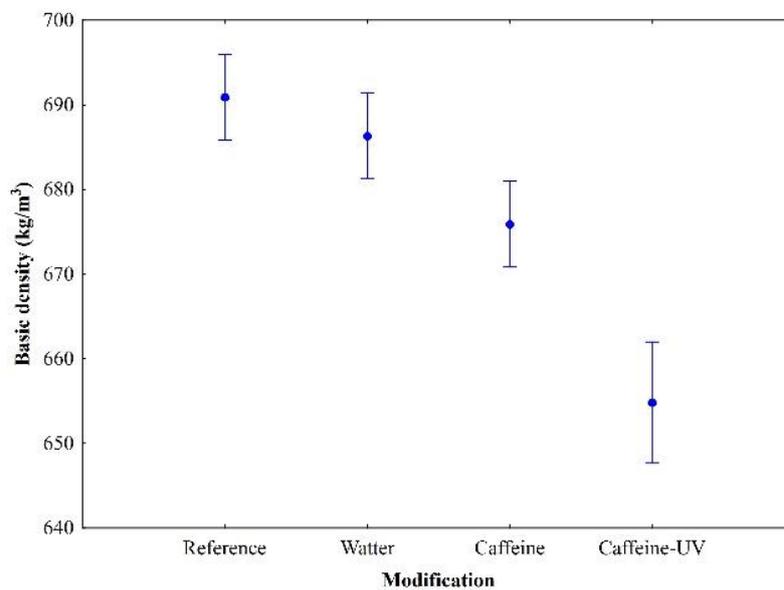
***Density***

Figure 1 shows an effect of modification on the oven-dry density. Although the effect of modification by caffeine and by soaking into distilled water was not statistically significant, decreasing trend is visible. It can be therefore assumed that caffeine solution has a certain negative effect on the density. Caffeine modification, followed by artificial weathering had significant effect on the density, not only from the view of statistical analysis but also from the view of industry.



*Figure 1: Effect of caffeine modification on oven-dry density*

Figure 2 shows the effect of modification on the basic wood density. The results are similar to those obtained for the oven-dry density. If we could speak about the trend in this case, the decrease in the density is steeper for modification by soaking into distilled water and by caffeine. In any case, the differences are insignificant. The effect of artificial accelerated weathering on the basic wood density of beech wood is again striking.



*Figure 2: Effect of caffeine modification on basic wood density*

### **Swelling**

Figure 3 shows the effect of modification on the volumetric swelling. Similar distribution of the properties, as in the case of density, was confirmed. Although the differences among reference samples, soaked into distilled water and modified by caffeine are not significant, the decreasing trend is apparent. Similarly to density, samples exposed to accelerated weathering are “out of scale”, showing not only statistically

significant difference but also low that should be taken into account when using such beech lumber for different purposes.

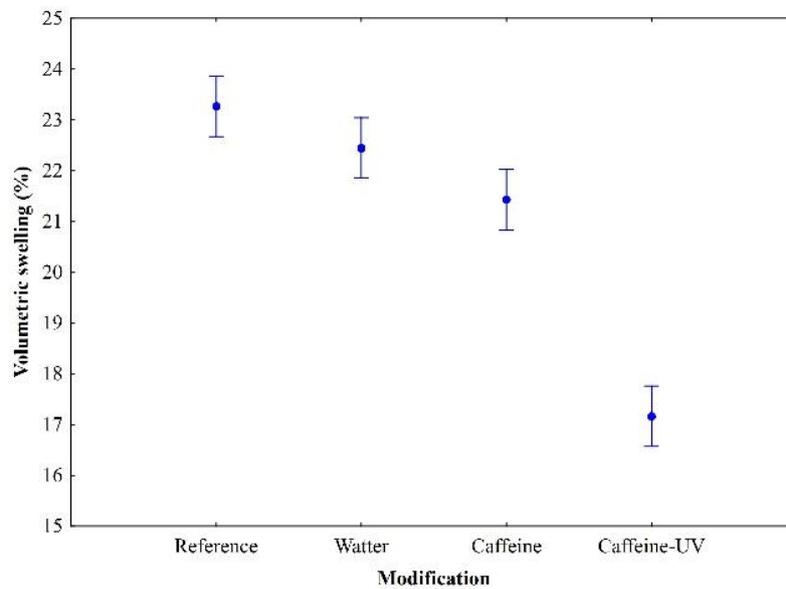


Figure 3: Effect of caffeine modification on volumetric swelling

### Compression strength

Figure 4 shows the effect of modification on the compression strength, the only representatives of the mechanical properties. Compared to the physical properties, this characterises was more variable. Nevertheless, although the differences among individual modifications were not significant, the same trend, as in the case of density, can be observed. It is natural because strength properties are closely correlated to wood density (Bodig and Jayne 1982, Dinwoodie 2000, Pożgaj et al. 1993).

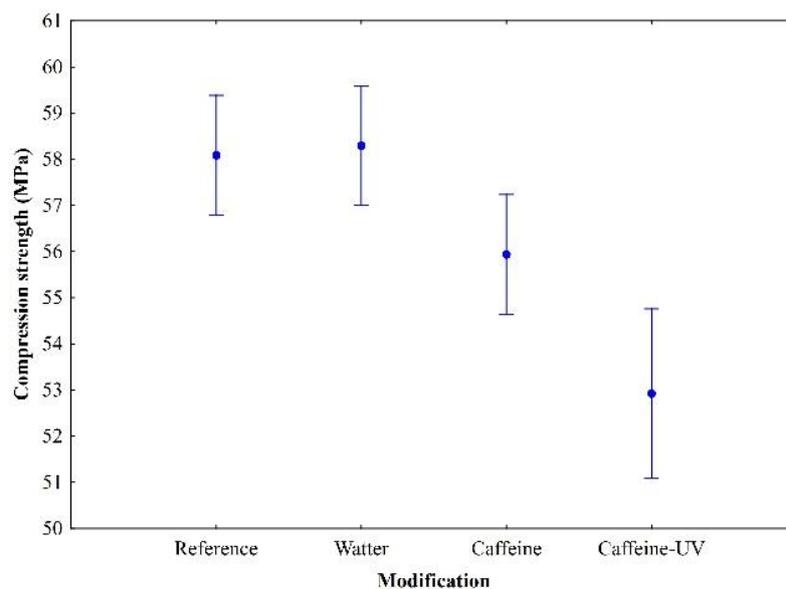


Figure 4: Effect of caffeine modification on compression strength

The decrease in wood properties for modification by caffeine is related to the soaking of samples in an aqueous solution containing caffeine, and thus to the possible leaching of substances without binding to the

structure of the wood. Furthermore, it should be noted that soaking in aqueous solution can no longer be considered as a purely viscoelastic deformation, as is the case with dimensional changes due to air humidity, but irreversible deformations also occur to some extent, which is logically reflected in lower swelling values (see water and caffeine), which is a well-known effect (Siau 1984, Skaar 1988). However, all the obtained data do not affect the overall effect, and thus we can speak about the "harmlessness" of caffeine modification on selected physical and mechanical characteristics of beech wood. The reduction in density and strength values can be considered clearly insignificant from a practical point of view and the intended use, and it is possible to focus on the positives that can be obtained by applying caffeine to wood. A further decrease in the properties (logically an increase in swelling) caused by UV radiation is also standard, corresponding to the influence of this factor on the properties of wood in general (Sandberg et al. 2017).

## CONCLUSIONS

The effect of caffeine treatment on selected physical and mechanical properties of beech wood was investigated in this experiment. Based on our results, we can conclude that such kind of modification has negligible impact on the tested properties. The effect of caffeine modification is very similar to the effect of soaking into distilled water. If we found any effect, it was more or less statistically insignificant, having no consequences for processing and utilisation of such wood. In any case, the positive fact is non-reduction of the commercial properties of wood by caffeine treatment. The negative effect of UV radiation is a standard that is also valid for untreated wood.

## ACKNOWLEDGMENTS

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## Effect of Fire retardants pre-treatments on the mechanical properties of Cement Bonded Particle Board made of Poplar hybrid I214

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**Keywords:** Curing agents, Fire Retardants, Poplar, Cement/Wood compatibility, Cement hydration.

### ABSTRACT

Wood-based buildings materials like cement-bonded particleboard (CBPB) have a growing market in central of Europe, which leads to continuous developments because of requirements, and regulations that put on global and national level. Over the years, researches improved the cement wood compatibility, mechanical properties and settings of curing time of CBPB with Pre-treatments but there was no effort to increase its the fire resistance especially that fire safety is importance aspect in the building and building materials. CBPB is classified with in B-s1, d0 classification of fire resistance MSZ EN 13501-1:2007+A1:2010. Using fire retardants as pre-treatments for wooden particles that be used in the CBPB could upgrade it to the A<sub>2</sub> or A<sub>1</sub> classification (MSZ EN 13501-1) but the fire retardants should not affect the primary properties of CBPB which are the mechanical properties. The objective of this paper is to investigate the effect of fire retardants pre-treatments: Disodium-tetra borate, Diammonium Hydrogen Phosphate and Disodium Hydrogen Phosphate on the mechanical properties of CBPB made of poplar hybrid (*Populus cv. euroamericana I214*). Portland cement CEM I 42.5 were used for CBPB production with curing additives like Sodium Silicate (SS) and Polydiallyldimethylammonium chloride (PDDA)+montmorillonite (MM). In total 16 type of CBPB were prepared 4 of them were control with untreated particles. After 15 days from CBPB's curing, specimens were cut with the required dimensions for each test. Results showed that fire retardants has influence on the mechanical properties of CBPB's. However, using 5% of SS or 20% of (PDDA+MM) the boards can full fill the standard requirements.

### INTRODUCTION

The inorganic-bonded materials have been appeared in early 1900s, Cement Wood Composites (CWC) are one of the mineral bonded products. CWC appeared in 1920 with wood wool cement board (WWCB) with low density of 400kg/m<sup>3</sup>, after it in 1930, wood chips cement board with 600 kg/m<sup>3</sup> were made. In 1960 coarse wood particle cement board was made with density range between 500 to 700 kg/m<sup>3</sup> followed by that the development of Cement Bonded Particle Board (CBPB) with high density range between 1250-1400 kg/m<sup>3</sup> (Stokke et al.2013). Between the 60's and 70's most of researchers focused on the effect of cement/wood proportion on WCP properties and there results was varied because of the used particle geometry, treatments, wood species, panel density and many other factors (Moslemi and Pfister 1986). In 1990, the cement wood wool board were developed more and its density increased to 900 kg/m<sup>3</sup>. with the beginning of the 21 century in 2000 wood strand cement board (WSCB) were produced with density of 1000-1100 kg/m<sup>3</sup> (Stokke et al. 2013). CWC are usually used as insulation material or construction material (Quiroga et al. 2016). For construction, cement-wood composite is used as panels, and in some recent studies cement-wood composites were used in the main structural elements of buildings, such as beams (Bej6 and Tak6ts 2005; Frybort et al. 2008). Because of the CWC strength properties, it is usually used for interior and exterior applications and for acoustic properties (e.g., highway sound barriers) (Na et

al. 2014). Gunduz et al. (2018) stated that cement-bonded particleboards with composite form is an effective application in term of acoustic outdoor noise barriers.

Fire resistance is important factor for building materials in general materials made of magnesium cement wood products are fire resistance materials (Zuo et al. 2018). Saval et al. (2014) tested the flammability of CBPB made of cement and Oceanic posidonia waste. CBPB is considered not a flammable material because no flame spread was occurred. According to the literature, the fire resistance of the cement-wood composites can be effected by the cement/wood ratio. A study was conducted on recycled Chinese fir particles and cement. The investigation was made by using a cone calorimetry test. Results showed that the cement-wood ratio had an influence on the fire resistance of the CBPB. With an increase in the cement-wood ratio from 0.5 to 2, the ignition time rised from 26 s to 548 s, and the mass loss rate decreased (Yu et al. 2016).

A number of studies have been performed on CWC for improving its shrinking and swelling, water absorption, and mechanical properties as well as increasing its producing time. However, less research was aimed at the fire resistance of the CWC. There was no wood pre-treatment investigation performed to improve the fire resistance of CWC, as was the case for increasing wood inhibitors. The only studies in this field concerned the non-combustibility of the material and the effect of wood ratio on fire resistance. CBPB is classified with in B-s1, d0 classification of fire resistance MSZ EN 13501-1:2007+ A1:2010 (Falco). Using fire retardants as pre-treatments for wooden particles that be used in the CBPB could upgrade it to the A2 or A1 classification (MSZ EN 13501-1) but the fire retardants should not affect the primary properties of CBPB which are the mechanical properties. The aim of this paper is to investigate the influence of different pre-treatment fire retardants on the mechanical properties of CBPB's made of Poplar hybrid I214 (*Populus cv. euroamericana I214*).

## EXPERIMENTAL METHODS

Derula ltd plywood company, (Magyarszecsöd, Szombathely, Hungary), supplied poplar (*Populus cv. euroamericana I214*) logs. Particles of Scots pine (*Pinus sylvestris*), Portland cement CEM I 42.5, and Sodium Silicate ( $\text{Na}_2\text{SiO}_3$ ) were obtained from Falco Zrt. wood industry company, (Szombathely, Hungary). Chemicals : Borax ( $\text{Na}_2\text{B}_4\text{O}_7$ ), DSHP ( $\text{Na}_2\text{HPO}_4$ ), DAHP ( $(\text{NH}_2)_4\text{HPO}_4$ ), montmorillonite (MM) ( $(\text{Na,Ca})_{0.33}(\text{Al,Mg})_2(\text{Si}_4\text{O}_{10})(\text{OH})_{2.n}\text{H}_2\text{O}$ ), Polydiallyldimethylammonium chloride (PDDA) ( $\text{C}_8\text{H}_{16}\text{NCl}_n$ ), were purchased from Thomasker Ltd (Budapest, Hungary)

### ***CBPB production, mat formation and processing***

Particles of poplar were treated with fire retardants by spray gun in a drum blender. The initial moister content of wood particle was around 18%. 1L of FR were sprayed for 3kg of wood.

CBPB were produced from treated poplar particles. The boards has three layer, the core layer with big particles and two surface layers with small particles (Fig.1). Sodium silicate was used as additive to increase the cement wood compatibility a mixture of PDDA and MM were used as well. The following recipes (Table 1) manufactured all 16 CBPB's.

***Table 1: CBPB recipes production.***

Recipe	R1	R2	R3	R4
Poplar particles	1	1	1	1
Cement	2.6	2.6	2.6	2.6
SS	0.052	0.13	0	0
PDDA	0	0	0.001	0.1
MM	0	0	0.001	0.1
Cement Stone	0.52	0.52	0.52	0.52



Figure 1: Seimpelkamp press, CBPB after 24 hour of pressing (right)

### ***Mechanical properties***

After 15 days, all produced CBPB's were cut for making standard tests.

### ***Bending test***

Test specimens with dimension (250x50x12 mm) were subjected to a load slowly to the point of failure with Instron test machine, the failure load, was recorded for each specimen, and modulus of rupture (*MOR*), modulus of elasticity (*MOE*) and density was calculated with Bluehill software attached to the test machine INSTRON IN5566 (EN 310).

### ***Internal bond test (IB)***

Test specimen with dimension (50x50x12 mm) were glued into two steel plates with dimension of (70x50x10 mm) under high pressure. A force perpendicular to the face were applied by INSTRON IN5566 test machine on test specimens gradually and continuously until reach failure and tensile strength were recorded (319).

### ***Thickness swelling test (TS)***

Thickness swelling was calculated as the percentage difference in length over the initial thickness (Eq.1). Test specimens with dimension (50x50x12 m) were immersed in water for 24 hours at room temperature 17°C. Thickness were measured at the middle of the specimen with digital thickness gauge (317).

$$TS = \frac{T_2 - T_1}{T_1} \times 100 \quad (1)$$

### ***Scanning electron microscopy (SEM)***

SEM images were taken for specimens with dimension (10X20X10mm) on x500 magnification, with accelerating voltage 20000V and  $v_{acc} = 20kv$  with Hitachi S-3400N instrument (Fig.2). SEM images were taken to compare the cement wood bonding for each specimen.

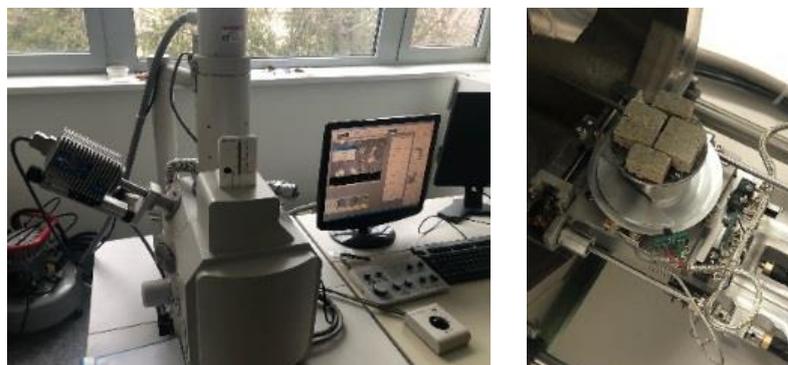


Figure 2: Hitachi S-3400N scanning electron microscopy (left), test specimen (right)

## RESULTS AND DISSCUSSION

For CBPB's made of poplar particles with 2 % of SS, board made of untreated wood particles did not fulfilled the standard requirement with MOR= 8.77 Mpa and IBS=0.48Mpa. Moreover, board made with treated particle has even lower MOR. Borax has decreased the MOR by 23.26 % and IBS by 29.16 %, while DSHP77g/l has decreased MOR by 16.10 % and IBS by 22.92 % for board with DAHP150g/l MOR decreased with 70.82 % and IBS by 64.58%. After adding 5% of SS from cement weight, MOR increased by 55.16 % for the untreated board MOR becoming 15.38 Mpa and IBS 0.68 % Mpa fulfilling the standard requirement. Boards made of treated particles, it's MOR increased by 51.96 % and IBS by 54.67% in case of board made of particles treated of borax, 35.92 % for MOR and 37.29 % for IBS in case of DSHP7g/l and 67.27 % for MOR and for IBS 71.18 % in case of DAHP150g/l. With using 5% of SS boards made of particles treated with borax and DSHP77g/l fulfilled the standard requirement while DAHP 150 stand out of standard see Table 2.

**Table 2: Properties of CBPB's made of Sodium Silicate as additives**

SS	Untreated	Untreated	Borax 2%	Borax 5%	DS 77g/l	DS 77g/l	DA	DA 150g/l
	2%	5%			2%	5%	150g/l 2%	5%
	Mean/SD							
MOR(Mpa)	8.77/0.84	15.38/1.18	7.36/1.04	12.76/1.02	5.18/0.96	9.29/0.91	3.01/0.86	6.47/1.33
IBS (Mpa)	0.48/0.03	0.67/0.05	0.34/0.02	0.75/0.03	0.37/0.10	0.59/0.04	0.17/0.03	0.59/0.03
Density (kg/m <sup>3</sup> )	1246.33 /31.81	1328.58 /51.41	1222.89 /63.40	1283.66 /56.92	1151.37 /50.43	1361.81 /21.88	1057.52 /53.11	1289.36 /77.29
TS (%)	4.78/0.62	3.33/0.54	2.62/1.70	2.61/0.25	4.12/1.02	3.35/0.90	1.96/0.53	0.69/0.52

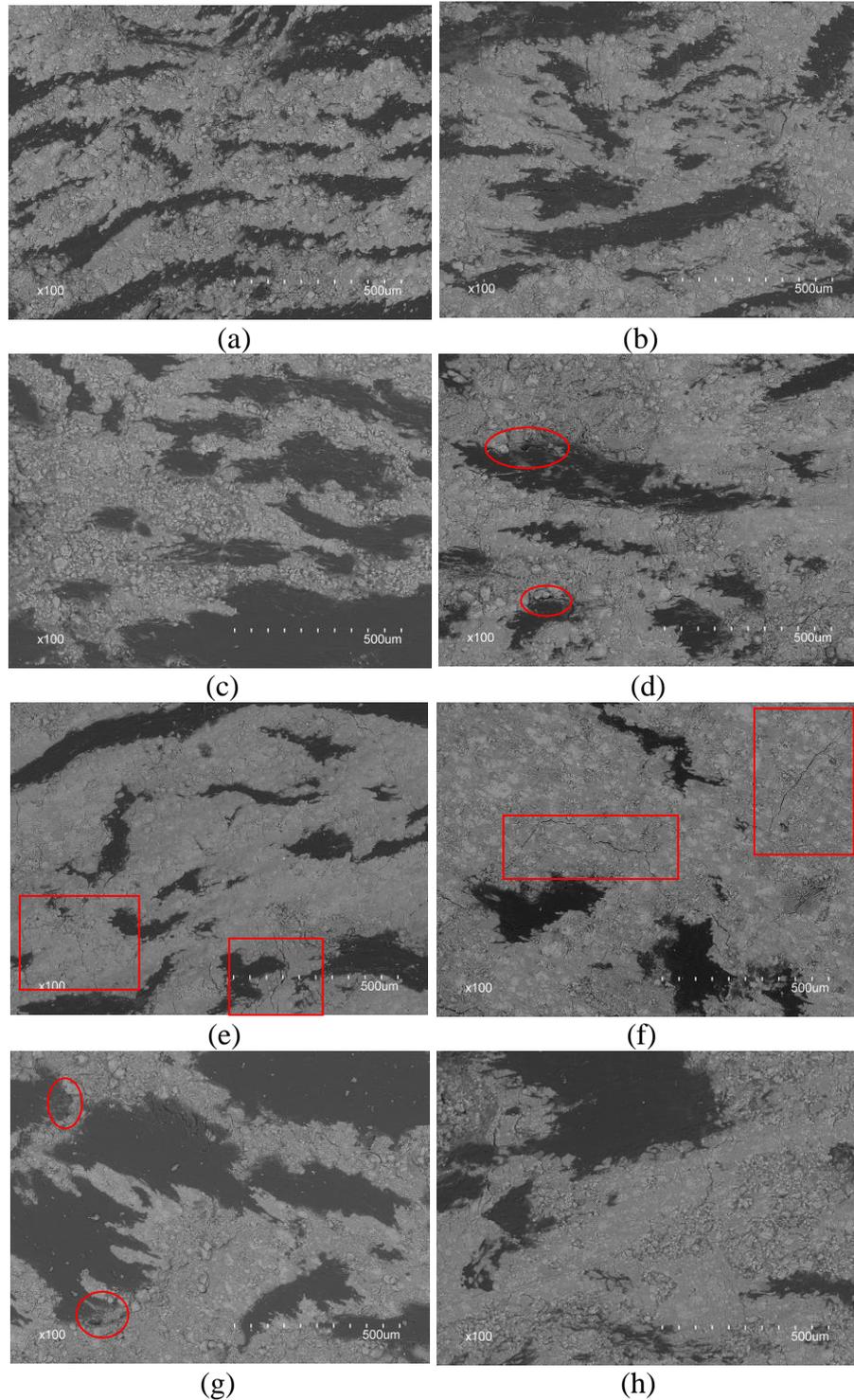
For boards made of poplar particles with 0.2% of (PDDA+MM), untreated board did almost met the standard requirement with MOR =8.64 Mpa and IBS= 0.28 Mpa. Fire retardants pre-treatments decreased MOR of the CBPB's for borax it was decreased by 78.82% for MOR and 12 % for IBS. DSHP77g/l it was decreased by 51.96 % MOR, 64.28 % IBS. MOR decreased by 50.06 % and IBS by 21.42 % in case of DAHP150g/l. By increasing the (PDDA+MM) to 20 % control specimens MOR increased by 49.01 % and IBS by 54.84 %, MOR of boards with DSHP77g/l pre-treatment increased with 58.22% and 82.47 % for IBS. MOR boards with DAHP150g/l pre-treatment increased with 53.31 % and with 67.65 % for IBS. Upgrading the boards to the standard requirements. However, for borax pre-treatment even with 20 % of (PDDA+MM) board cured but have low MOR 4.19 Mpa and IBS 0.45 Mpa see Table 3.

**Table 3: Properties of CBPB's made of PDDA+MM as additives**

PDDA+M M	Untreated	Untreated	Borax	Borax	DS 77g/l	DS 77g/l	DA150g/	DA150g/
	2%	5%	2%	5%	2%	5%	12%	15%
	Mean/S	Mean/SD	Mean/S	Mean/S	Mean/S	Mean/SD	Mean/S	Mean/S
MOR	8.64/1.6	16.94/1.0	1.83/0.2	4.19/1.2	4.49/0.6	10.75/2.5	4.31/0.46	9.24/0.93
IBS (Mpa)	0.28/0.0	0.62/0.07	0.25/0.0	0.45/0.0	0.10/0.0	0.57/0.48	0.22/0.03	0.68/0.10
Density (kg/m <sup>3</sup> )	1338.27 /77.86	1375.96 /15.88	1020.03 /59.13	1161.02 /49.19	1198.89 /36.19	1329.37 /110.03	1194.46 /61.13	1249.81 /46.99
TS (%)	6.81/2.7	2.84/0.58	2.98/0.7	1.29/0.4	5.54/1.7	3.46/0.80	4.65/1.89	0.77/0.27

Increase in the bond agent concentration leads to increase in MOR and in the same time increase in the CBPB's density. CBPB's made of particles pre-treated with borax has the low density with high MOR comparing with other CBPB's in case of using SS as bond agent. For thickness, swelling increasing the concentration of bond agents reduce the TS of the boards, best results were achieved by CBPB made with particles pre-treated with DAHP 150g/l on which TS was reduced by 85.56% by using SS as bond agent and 88.69% by using PDDA+MM see Table 1 and Table 2.

Specimens from boards made with high concentration of bond agents were taken and Scan electron microscopy (SEM) images were taken for comparing the CBPB's with each other see (Fig.3).



**Figure 3: SEM images for CBPB's. (a) Untreated CBPB with SS. (b) Untreated CBPB with PDDA+MM. (c) Borax CBPB with SS. (d) Borax CBPB with PDDA+MM. (e) DSHP77g/l CBPB with SS. (f) DSHP77g/l CBPB with PDDA+MM. (g) DAHP150g/l CBPB with SS. (h) DAHP150g/l CBPB with PDDA+MM.**

Comparing specimens with each other, untreated CBPB's for both SS and PDDA+MM see Fig.3 (a-b) wood particles are compatible with cement mixture the colour of the cement paste and shape indicate that the cement hydrated very well. In other hand, for CBPB's made of Borax-PDDA+MM and DAHP 150g/l-SS shown low compatibility between cement and wood particles in many parts the cement wood mixture was separated from wood particles its shown by red circles in Fig.3(d-g). CBPB's made of DSHP 77g/l has many cracks which represented by squares in Fig.3 (e-f). The cracks results on fast drying of the cement

wood mixture. That leads to conclusion that DSHP 77g/l drying the mixture, and when looking to thickness swelling of CBPB made of DSHP 77g/l it has very high TS.

## CONCLUSION

Over the years, many scientists has been Improved CWC from mechanical properties, cement wood compatibility to shortening production time via pre-treatments. However, enhancing the fire resistance of CBPB with fire retardants pre-treatment was never took into considerations. This paper studied the effect of fire retardants pre-treatments on the properties of CBPB for use it later to the possibility of improving fire resistance of CBPB made poplar particles pre-treated with fire retardants. It was found that fire retardants has big influence on the MOR and IBS of the CBPB's but with using 5% of SS CBPB's pre-treated with borax or DAHP77g/l fulfill the standard requirements. In other hand, using 20% of (PDDA+MM) for producing CBPB's pre-treated with DAHP 150g/l or DSHP 77g/l fulfill the standard requirements. Using DAHP 150 g/l can reduce the TS by more than 80%.

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## Impacts of a tree species shift on the Austrian forest-based sector

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**Keywords:** forest-based sector, forest sector model, tree-species shift, seedling demand, climate change

### ABSTRACT

The forest-based sector and the increased use of wood products can make an important contribution to climate change adaptation and mitigation. Wood products not only store carbon from the forest in an additional carbon cycle but also help to substitute fossil-based/abiotic products in material and energy use. The efficient circular or cascade use of wood helps to keep carbon longer in the product-based carbon cycle. The presented study compared potential effects of a tree species shift through adaptive forest management (“tree shift scenario”) in a simulation-based scenario analysis with a business-as-usual scenario (“reference scenario”). Mast seeding predictions and selected time series with a potential influence on seedling demand (logging activity, timber prices, and economic indicators) were used to formulate multivariate econometric relationships between mast occurrence and seedling demand. Subsequently, a dynamic lagged partial equilibrium model representing the Austrian forest-based sector encompassing forest resources, raw material markets and semi-finished product markets including trade with a rest-of-the-world region was used to thoroughly investigate interdependencies within sub-sectors of the forest-based sector. The model was coupled with an individual-tree-based growth model with data being exchanged iteratively on an annual basis through a cloud-based solution. This approach allowed to simulate the demand for seedlings for seven main tree species based on market framework conditions for seedling demand for spruce, fir, larch, scots pine, stone pine, oak, and beech for both scenarios. For wood utilisation, species data was aggregated for coniferous and non-coniferous wood. The time horizon for the simulations was 2100 to consider effects well beyond the average rotation period.

### INTRODUCTION

Climate change (CC) will considerably affect local environmental conditions for Central European tree species. This will have significant impacts on various ecological and economic conditions, because stand-forming forest trees often dominate and shape complete ecosystems thereby guaranteeing ecosystem functioning, carbon storage (Stümer et al. 2010) and various benefits for the human population (Mantau et al. 2001; Hanewinkel et al. 2013). CC will strongly affect the seedling demand of the majority of native and non-native tree species if Austrian forest owners adapt forests to CC. This seedling demand needs to be considered by nursery and forest seed managers when planning future growing stock and sales.

Besides medium- and long-term adaptation measures to CC, the seedling demand for the various tree species depends on various economic factors which are partly interdependent with ecological conditions and also influenced by political framework conditions, actions set by decision and policy makers and interdependencies with the global economy. Demand for raw-material is furthermore shaped by strategic decisions executed by forest-based industries reacting on the forest products market. However, forest-based industries have to adapt to available raw-material supply and try to react to exogenous economic conditions such as impacts of the global market on foreign trade (Hajdúchová et al. 2016), prices from potential trade partners, potential for increased efficiency and product diversification, oil prices, the state of the national

economy and as a consequence the labour market (Näyhä et al. 2015, Lehtonen and Okkonen 2013). Proactively envisaging adequate action for adaptive forest management through investigating the seeds and seedlings needed for the future treads the path to ensure resilience of all involved branches of the forest-based sector. Nursing seeds and providing seedlings is connected with considerable financial expenditure and great risks because it is a long-term operation which cannot react to short-term fluctuations of the markets. It can nevertheless adapt to climatic trends and general requirements of the forest-based sector when the interdependencies and requirements are analysed comprehensively. Through dynamic forest sector models, it is furthermore possible to adapt to a bandwidth of possible futures with respect to the national economy and various branches of forest-based industries.

## METHODS

Historical data on mast seeding and seed harvest from 1960 to 2016 were collected and analysed for seven native tree species – Norway spruce (*Picea abies*), silver fir (*Abies alba*), European larch (*Larix decidua*), Scots pine (*Pinus sylvestris*), Swiss stone pine (*Pinus cembra*), European beech (*Fagus sylvatica*), Oak (aggregate of *Quercus robur* and *Quercus petraea*) – with the aim of investigating the link between local climate conditions and the synchronised occurrence of large amounts of fruits and seeds at irregular intervals (mast seeding). Reports on mast seeding from 1962-1995 for eight stand-forming species in Austria were analysed. Machine learning methods (ADA, Adaptive Boosting; DT, Decision Tree; GB, Gradient Boosting; LR, Logistic Regression; MLR, Multiple Linear Regression; RF, Random Forests; SVR, Support Vector Regression) were utilised to determine mast association with climatic variables and to conduct projections of mast occurrence until 2100 for two scenarios (using EURO-CORDEX-data for RCP 4.5 MPI-ESM-LR/RCA and RCP 8.5 MPI-ESM-LR/RCA).

Data was collected to create a consistent database of seed harvests as well as for potential factors determining the supply and demand of seedlings for the seven target species. Data collection was completed for the period 1991-2016 thus providing annual data for 26 years. Until 2011/12, production volumes were reported by seedling producers based on estimates of their current production volumes. Since 2012/13 the collection of this data set has been discontinued. However, the production data for the years 2012/13-2016/17 were completed in March 2018 based on a new methodology. Producers indicate the sales figures of seedlings in relation to their seed origin, which leads to much more precise data. The sales figures are lower than previous production data because the data collection is based on real data. In addition, old data on seed harvest reports (before 1997) had to be digitised and aggregated annually. Data for seedling production/sales (subsequently denoted as seedling production) was aggregated with dummies representing the change in data collection.

In a next step cross correlation analyses were performed to investigate the influence of mast occurrence on seedling production because mast occurrence was the only available forest ecological indicator potentially influencing seedling availability. Additionally, correlation analyses were conducted for forest-based sector related time series with a potential influence on seedling production (such as logging activity, timber prices, and economic indicators). Subsets were selected based on correlation results, plausibility and availability (either because future simulation for respective variables are available from the subsequently used forest sector model or because long-term projections are available).

Based on the subset, stepwise regression was applied to select regressors resulting in the highest model adjusted R<sup>2</sup> with a limit of maximum six estimators. For each species regressions were performed with absolute values. They can be formulated as:

$$Y_t = \alpha_0 + \beta_1 * x_{1t-\tau} + \beta_2 * x_{2t-\tau} + \dots + \beta_n * x_{nt-\tau} + \epsilon \quad (1)$$

where:  $Y$  ... dependent variable (e.g. yearly seedling production/sales of silver fir)  $\alpha_0$  ... constant  $x_1, x_2, \dots, x_n$  ... explanatory variables (e.g. coniferous logged wood)  $\beta_1$  ... coefficients of explanatory variable  $\tau$  ... number of lags.

Subsequently, regression equations were implemented into the model of the Austrian forest-based sector (FOHOW, “Forst- und Holzwirtschaft”), a dynamic (lagged) partial equilibrium model formulated following the System Dynamics methodology. It represents the entire value chain of the Austrian forest-based sector as an interconnected system encompassing forest resources, raw material markets and semi-

finished product markets including trade with a rest-of-the-world region (for details cf. Schwarzbauer 1993, Braun et al. 2016). Simulations of logging activity were conducted iteratively (exchanging annual data through a cloud-based solution) coupled to an individual-tree-based growth model developed by the Federal Research and Training Centre for Forests (BFW). For the forest sector model it was assumed that with moderate CC (RCP 4.5) a business-as-usual approach will guide forest management (*Reference scenario*) while with strong CC (RCP 8.5) adaptive forest management will favour a gradual substitution of spruce with seedlings from more adequate provenance (*Tree shift scenario*) Thus the demand for seedling could be simulated in dependence of the dynamic evolution of model-endogenous variables and externally projected variables.

It is important to note that the simulated scenarios are NOT forecasts, but rather the mapping of relationships that are valid under certain “what-if” assumptions. These in turn are subject to very specific exogenous assumptions made, which only have a validity applicable to the scenario.

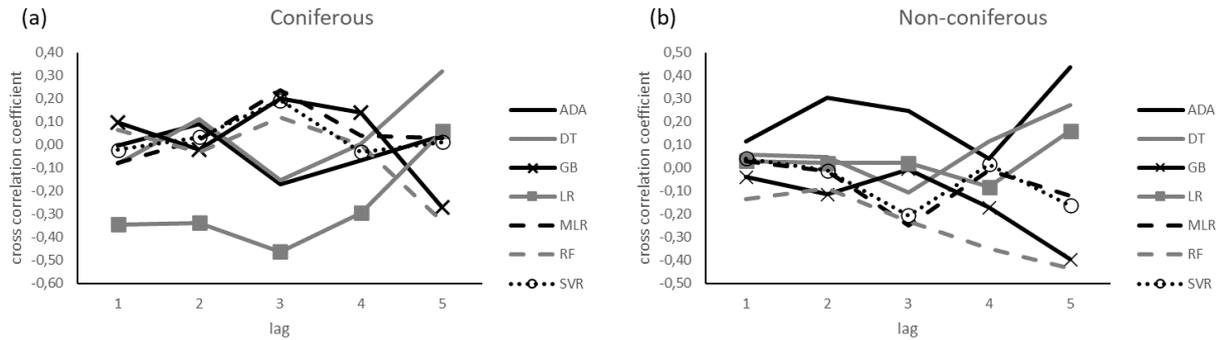
## RESULTS AND DISCUSSION

Generally, production numbers show a decreasing trend since the 1990s, both for coniferous and deciduous seedlings. Production numbers between 1991 and 2011 show a decrease of 34% for coniferous seedlings and 43% for deciduous seedlings. These trends can be attributed to several reasons. Logging activity, wood prices, seed provision and the macroeconomic situation are drivers identified. Furthermore, various trends affecting the demand for seedlings could not be adequately quantified. Clear cutting has been increasingly substituted by single stem removals. This change in forest management practices with a shift towards natural regeneration lowers the demand for forest seedlings. Furthermore, production and sales quantities of Spruce seedling quantities show a declining trend since 1991. As spruce exhibits by far the highest production quantities of all species, the decline for coniferous seedlings is presumably lower than represented in the production figures, but still existent. The overall share of spruce in Austrian forests only experienced a minor decrease since the 1980s (from 62% to 60% at the latest forest inventory in 2007/09). Pure spruce stands were especially affected by storm-induced damage such as in 2007 and 2008 (Russ 2011), and bark beetle infestation. However, the decrease of pure spruce stands from 44% to 38% over the last decades (from 1992/96 to 2007/09) shows that spruce is increasingly distributed across mixed stands. The steady share of spruce in Austrian forests in combination with a decreasing trend in spruce seedling production indicates that it is increasingly reproduced by natural regeneration. Despite ecologically problematic monocultural stands and poor rejuvenation efforts, spruce is still an important species to forest owners for economic reasons. However, they have become less important (Karopka 2017) and are increasingly substituted by larch, fir and broadleaves (Baschny and Strohschneider 2012). Furthermore, since 2011 sales numbers of fir, Scots pine and Swiss stone pine have been rising. Production numbers for deciduous species are much more volatile due to shorter production periods (1-3 years vs. 2-5 years). Production numbers for oak and beech have been decreasing since 1991. However, sales numbers since 2012 show an increase for oak and beech. Overall, sales quantities between 2012/13 and 2016/17 show a clear trend towards non-coniferous and mixed forests seedlings.

### *Correlation analysis*

Correlation analysis was conducted for data on tree species level. Due to availability, seed harvest data is the only (forest ecology-based) time series available for the whole period from 1991-2016 besides the target variable “seedling production”. Therefore, correlation analysis was performed between these two time-series for all tree species. The analysis includes lag structures, in order to highlight delayed correlation over time. Cross correlation with lag structure considered possible delayed effects of (Austrian) seed harvests on seedling production.

The starting hypotheses were that cross correlation is stronger for species with a high share of domestic genetic origin, and for species with a low maximum storage life of seedlings. When comparing the figures depicting cross correlations for aggregated coniferous and non-coniferous species there is a difference between the method used to model mast occurrence: For ADA, DT and LR the cross correlation is tendentially higher for non-coniferous species, while for MLR, RF and SVR the cross correlation is tendentially higher for coniferous species (Fig. 1).



**Figure 1: Cross correlation between mast occurrence and (lagged) seedling production in relation to mast occurrence predictions using ADA, DT, GB, LR, MLR, RF, SVR (abbreviation explanation cf. Methods section). (a) grouped means for coniferous species, (b) grouped means for non-coniferous species**

A more detailed look at the species level shows more ambivalence. For many species such as beech, scots pine, pedunculate oak only 1-2 significant positive (under the assumption that high seed occurrence must lead to high seedling occurrence) correlations could be found, with tendentially higher correlations for difference values (as in comparison to correlations for absolute values). The scarcity of data for seed harvest quantities for non-coniferous species affects the quality of predictions from models for mast occurrence. Generally, further investigation and discussion is needed on this topic in order to determine connections between mast occurrence (or seed harvest) and seedling production and their implications.

### Regression analysis

The regressions were run with both absolute values and first differences for the seven target species. All available data from 1991-2015 was considered. The year 2012 in the differenced time series is NA due to the change in methodology. Because of the limited length of the time series, verification of the models is performed for the last year of the time series based on the estimation period. The last data point in the year 2016 is withheld from the estimation and instead calculated based on the selected predictors from the in-sample-estimation. Out-of-sample validation is crucial and shows the model's ability to produce accurate and stable predictions. Validations can highlight potential problems of model specifications such as overfitting or selection bias. As expected, regressions with absolute values show higher (adjusted)  $R^2$  values and lower p values for estimators (with exception for Oak, since most coefficients for the Oak model for absolute data are not significant). The long-term correlations are captured in the models and translated into results with higher significance.

**Table 1: Regression results for seedling demand for non-coniferous tree species (oak and beech); variables explained under Table 2**

Non-coniferous									
Oak (adj. $R^2=0.57$ )					Beech (adj. $R^2=0.77$ )				
Term	Estimate	Std. Error	t value	Pr(> t )	(Intercept)	-1,71E+02	1,83E+02	-0,94	0,36
(Intercept)	-4,44E+02	6,87E+02	-0,65	0,53	bu_ADA3	1,71E+02	1,34E+02	1,27	0,22
eil_GB3	1,99E+02	2,34E+02	0,85	0,40	bu_ADA5	1,71E+02	1,49E+02	1,15	0,27
oebf_l3	-7,61E-03	4,09E-03	-1,86	0,08	dummy_abs	3,80E+02	8,61E+01	4,41	0,00
oebf_end_l2	2,72E-03	2,67E-03	1,02	0,32	oebf_end_l2	1,45E-03	7,05E-04	2,05	0,06
oebf_end_l3	1,18E-02	5,13E-03	2,30	0,03					

Based on the seven selected tree species seven regression equations were selected based on absolute values and seven based on relative values. Tab. 1 (non-conif.) and Tab. 2 (conif.) show selected regression equations with the combinations of variables identified to be most significant for coniferous and non-coniferous species respectively. In the stepwise selection process regression equations were selected by highest adjusted.  $R^2$  for sensible coefficient combinations with a limit of maximum six coefficients. Overall, results show that logging activity is the most important estimator by contributing to all but two regression estimations (in various variations), covering up to 4 out of the 6 possible time-shift combinations (0-5-year

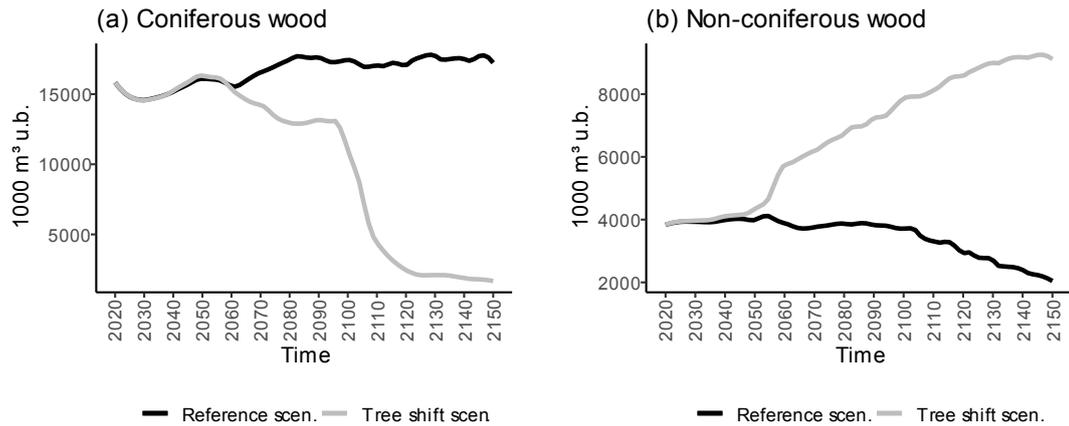
lag). Because in the next step (forest sector modelling) regression for first order differences did not yield meaningful results, they are not depicted.

**Table 2: Regression results for seedling demand for coniferous tree species; variables explained under Table 2**

Coniferous									
Spruce (adj. R <sup>2</sup> =0,80)					Fir (adj. R <sup>2</sup> =0,85)				
Variable	Estimate	Std. Error	t value	Pr(> t )	Term	Estimate	Std. Error	t value	Pr(> t )
(Intercept)	-4,81E+04	1,37E+04	-3,50	0,00	(Intercept)	-8,57E+02	7,83E+02	-1,09	0,29
fi_MLR1	1,32E+03	1,18E+03	1,12	0,28	ta_SVR1	7,73E+01	1,05E+02	0,74	0,47
dummy_09	-1,01E+04	3,95E+03	-2,56	0,02	dummy_09	2,34E+03	3,71E+02	6,32	0,00
dummy_abs	1,85E+04	3,70E+03	4,98	0,00	hem_end_ges1	-1,36E-03	4,67E-04	-2,91	0,01
oebf_ges	-2,06E-02	1,20E-02	-1,73	0,10	hem_end_n1	1,43E-03	5,16E-04	2,76	0,01
oebf_end_ges	2,43E-02	1,22E-02	1,99	0,06	oebf_end_ges2	5,32E-04	2,15E-04	2,48	0,02
FiTa_pwd_mix1	1,79E+03	2,35E+02	7,61	0,00	FiTa_pwd_mix2	7,63E+01	1,87E+01	4,07	0,00
Larch (adj. R <sup>2</sup> =0,91)					Scots pine (adj. R <sup>2</sup> =0,75)				
Term	Estimate	Std. Error	t value	Pr(> t )	Term	Estimate	Std. Error	t value	Pr(> t )
(Intercept)	-1,49E+04	1,76E+03	-8,46	0,00	(Intercept)	-4,29E+03	7,14E+02	-6,01	0,00
la_MLR3	3,37E+02	2,73E+02	1,23	0,24	ki_ADA1	-7,35E+02	3,71E+02	-1,98	0,07
dummy_09	-1,64E+03	6,00E+02	-2,74	0,01	ki_ADA3	1,47E+03	4,05E+02	3,63	0,00
dummy_abs	3,90E+03	8,98E+02	4,35	0,00	dummy_abs	7,65E+02	1,50E+02	5,09	0,00
gr200_n3	4,32E-04	3,66E-04	1,18	0,26	Kie2a4	2,04E+01	9,32E+00	2,19	0,05
FiTa_pwd_mix	1,00E+02	5,51E+01	1,82	0,09	Kie2a5	4,87E+01	8,75E+00	5,57	0,00
fwd_conif1	3,17E+02	7,36E+01	4,31	0,00	gdp_gr_real	-9,79E+01	3,60E+01	-2,72	0,02
Stone pine (adj. R <sup>2</sup> =0,77)									
Term	Estimate	Std. Error	t value	Pr(> t )					
(Intercept)	-1,89E+03	3,19E+02	-5,91	0,00					
zi_DT3	4,10E+02	1,38E+02	2,98	0,01					
oebf_ges2	1,68E-03	3,74E-04	4,47	0,00					
oebf_end_ges2	-1,31E-03	3,68E-04	-3,56	0,00					
Kie2a5	6,48E+00	3,42E+00	1,89	0,08					
gdp_gr_reall	7,01E+01	1,48E+01	4,76	0,00					
Abbreviations used in Table 1 and 2									
FiTa_pwd_mix1... price spruce/fir pulpwood mixed assortment price (lag 1 year)					gr200_n3... total logging forest enterprises (>200 ha) (lag 3 years)				
FiTa_pwd_mix2 = " (lag 2 years)					hem_end_ges1... total final fellings (lag 1 year)				
Kie2a4... price for pine, category B 2a+ (lag 4 years)					hem_end_n1... total final fellings of nonconiferous wood (lag 1 year)				
Kie2a5... " (lag 5 years)					ki_ADA1... Scots pine mast prediction, adaptive boosting (lag 1 year)				
bu_ADA3... beech mast prediction, adaptive boosting (lag 3 years)					ki_ADA3... " (lag 3 years)				
bu_ADA5... " (lag 5 years)					la_MLR3... larch mast predictions, multiple linear regression (lag 3 years)				
dummy_09... dummy variable, unexplained peak in seedling production in 2009					oebf_end_ges... final fellings state forests				
dummy_abs... dummy variable for change in reporting production vs. sales (cf. Methods)					oebf_end_ges2... " (lag 2 years)				
ei1_GB3... oak mast prediction, gradient boosting (lag 3 years)					oebf_end_l2... final fellings state forests (lag 2 years)				
fi_MLR1... spruce mast prediction, multiple linear regression (lag 1 year)					oebf_end_l3... final fellings state forests (lag 3 years)				
fwd_conif1... price coniferous fuelwood (split logs) (lag 1 year)					oebf_ges... total logging state forests				
gdp_gr_reall... relative change in real GDP (lag 1 year)					oebf_ges2... " (lag 2 years)				
					oebf_l3... total logging state forests (lag 3 years)				
					ta_SVR1... fir mast prediction, support vector regression (lag 1 year)				
					zi_DT3... Swiss stone pine mast prediction, decision tree (lag 3 years)				

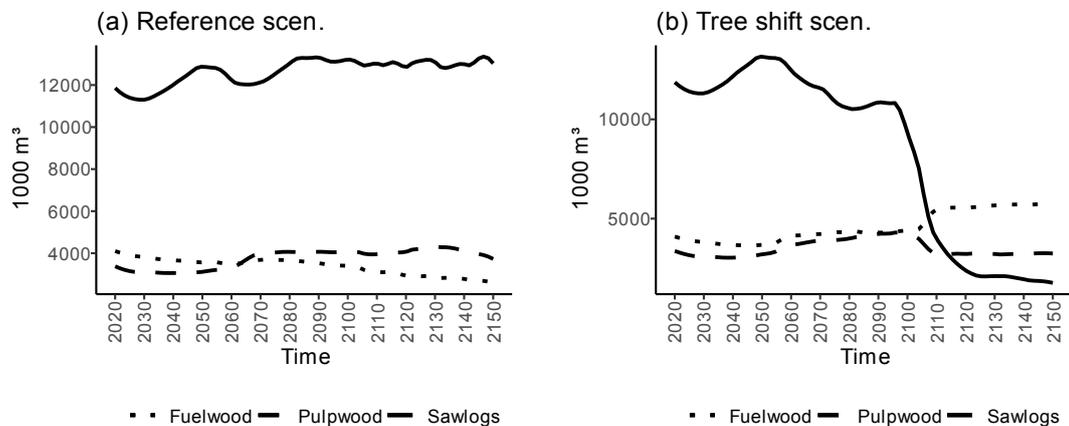
### Simulation

Fig. 2 shows the development of wood supply for both scenarios for coniferous (Fig. 2a) and non-coniferous wood (Fig. 2b). While it is possible to safeguard wood supply over a long period of time in the reference scenario, adaptive forest management in the *Tree shift scenario* induces strong changes in forest communities to reduce the amount of damaged wood which is expected to increase continuously throughout the simulation period with considerable increases from about 2070. Because potential adaptation of forest-sector industry and enterprises was not included in the modelling efforts, the demand for non-coniferous wood other than for fuel wood is still low leading to a dramatic reduction of overall wood use. This is an important illustration to show that mid- to long-term adaptation of forest industry and enterprises is important to safeguard the functioning of the Austrian wood products market. While the increased availability also leads to an increase in wood supply, most of it is used for energy purposes.



**Figure 2: Development of wood supply from Austrian forests with and without tree species shift; (a) coniferous wood, (b) non-coniferous wood**

Fig. 3 shows the development of fuelwood, pulpwood and sawlog availability for the *Reference scenario* (Fig. 3a) and the *Tree shift scenario* (Fig. 3b). While in the *Reference scenario* there is decreased wood availability until 2050 (mainly caused by reduced availability of wood from thinning activities), long-term availability stays rather constant until the end of the simulation period. There is increased inter-sectoral competition to some extent favouring material use of wood leading to a reduction in fuelwood availability. The *Tree shift scenario* shows the described reduction in total pulpwood and sawlog production due to the non-demand of forest processing industry for non-coniferous wood which is also visible in increased fuelwood consumption due to low prices.



**Figure 3: Production of fuelwood, pulpwood and sawlogs aggregated for coniferous and non-coniferous wood. (a) reference scenario, (b) tree species shift scenario**

Results for seedling demand (Fig. 4) show that spruce will be still in high demand in the *Reference scenario* (if current forest management practices and species use can be continued). For the *Tree shift scenario*, the demand for spruce seedlings is expected to decline from about 2030 and to significantly drop from 2050 (Fig. 4a). Fir seedlings are expected to slightly increase in demand from about 2050 in the *Reference scenario* while the *Tree shift scenario* projects an increase in demand by mid-century – probably to substitute spruce trees (Fig. 4b). Larch and Scots pine seedlings are expected to increase in demand in both scenarios as a part of adaptive forest management (Fig. 4c-d). They are still preferred by the wood processing industry which is heavily specialised on softwood in Austria. While Swiss stone pine plays

rather a niche role, it is expected to increase in demand, probably due to continued suitability in alpine and peri-alpine regions (Fig. 4e).

Beech (Fig. 4f) and oak (not depicted) will increase in demand due to expected forest management requirement. In both scenarios oak, and to a lesser extent beech are strongly increasing in demand. While in the *Reference scenario* there is a strong increase in demand until about 2050, further demand is expected to stay constant, while in the *Tree shift scenario* oak seedlings are in high demand until about 2050 with the demand subsequently being substituted by increasing Beech seedling demand.

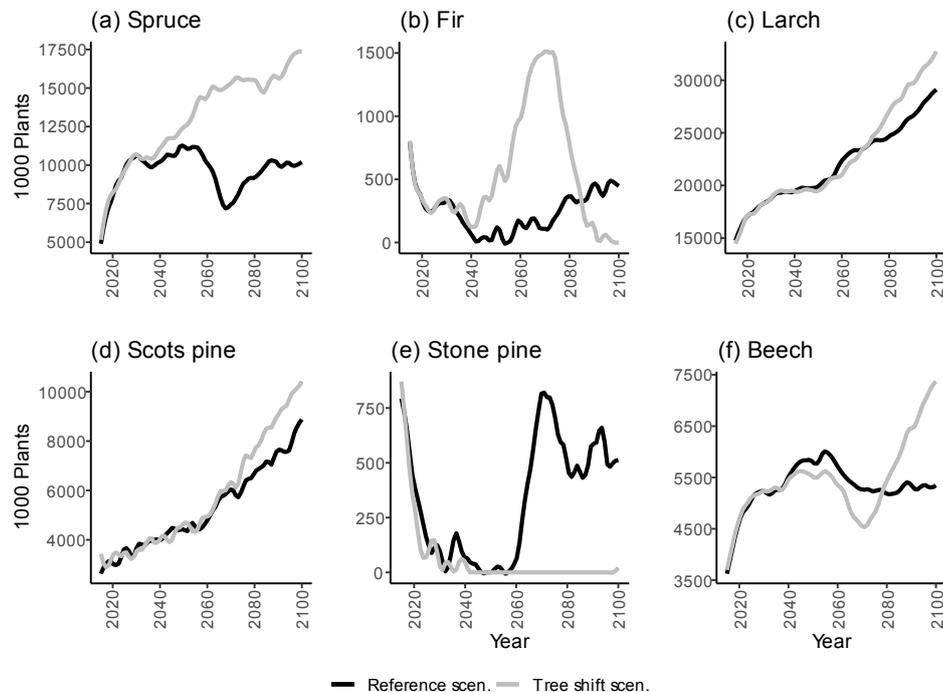


Figure 4: Simulated results for seedling demand depending on forest sector framework conditions; (a-f) by tree species (Oak omitted)

## CONCLUSIONS AND OUTLOOK

The simulations for wood availability and seedling demand for both scenarios are both modelled with an assumed non-adaptation of the wood processing industries and enterprises to illustrate the effects on the entire forest-based sector as well as the carbon dynamics (not discussed in the paper at hand). The demand for seedlings is heavily dependent on adaptation measures not only in the forest but in the entire wood-based value chain. Innovation management will play a crucial role in shaping future seedling demand and can change the pathways projected in the scenarios at hand. To expand this view, it is important to develop anticipative innovation models with stakeholders (e.g. through stakeholder-centric approaches such as collaborative conceptual modelling as used in health research cf. Friel et al. 2017 and through understanding what drives innovation, i.e. the (micro-)foundations of companies' dynamic capabilities for innovation cf. Teece 2007)

Future research endeavours will aim to include additional forest ecological and climatic variables and to expand the dataset of long-term forecasts for variables connected to forest management (e.g. clear cut areas) to increase the predictive quality of the models (and to make first order difference value models useful). An adaptation of the Austrian forest industry and enterprises was not modelled. It is of cardinal importance to research (and to provide funding for) the investigation of future innovation pathways in the forest-based sector, especially in forest products modelling to better understand the reaction of the economy to tree species shifts/to forest management adaptation measures e.g. through stakeholder-centred collaborative conceptual modelling efforts.

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## **Beech for structural purposes – a case study in south-west Germany**

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**Keywords:** Beech timber, structural timber, roundwood quality, structural timber quality

### **ABSTRACT**

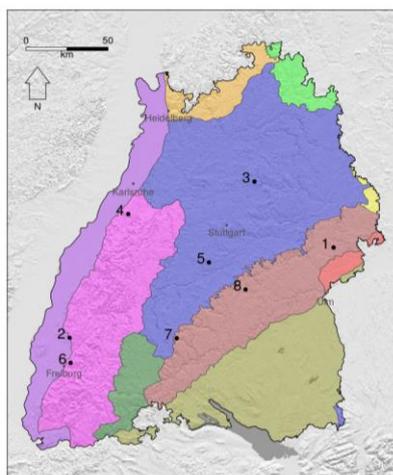
Small-diameter beech roundwood from 8 different stands in Baden-Württemberg (south-west Germany) (40 trees, 108 logs) varying in altitude and tree age was characterised by log quality and converted to boards for structural use. Results reveal a general trend of higher wood stiffness for material from lower altitude up to 23 kN/mm<sup>2</sup> for individual boards due to tree growth. However yield in sawn boards meeting structural requirements according to applicable regulations was generally low with about 30%. Boards were mainly rejected due to the presence of pith. The results are in line with other investigations on use of small diameter hardwood for structural purposes.

### **INTRODUCTION**

Recurring forest calamities in the second half of the 20th century caused a rethinking in the German forest industry, towards more diverse forest types and the promotion of hardwood species. As a consequence, the stock of hardwoods, especially of beech, increased while the stock of softwood declined. Thus there will be an increased supply of beech timber from thinnings which means timber in smaller diameters and poorer stem quality. The objective of this study is to investigate the potential of small diameter beech timber for structural purposes.

### **MATERIAL AND METHODS**

108 logs were sampled from 40 trees located on 8 sites in south-west Germany (Fig. 1, Table 1). The sites were chosen from different growth areas with a variation in altitudes. The dbh varied between 28 cm and 38 cm. The trees were cut into logs of 4,0 m to 5,0 m, with one to four logs per tree depending on tree height (Table 2).



**Figure 1: Map of Baden-Württemberg with location of the sites as presented in Table 1**

All logs were subject to visual grading according to German grading standard RVR (DFWR&DHWR 2015) (all criteria separately for further analysis), scanning with a Microtec<sup>®</sup> CT.Log scanner for log density measurement, and application of the ViScan tool (Microtec<sup>®</sup>) for measurement of the log's longitudinal swaying frequency and the dynamic log MOE (Fig. 2). After sawing, the subset of 628 edged, full-length boards (Table 1) (with a thickness of 35 mm and width of 90, 130, and 160 mm, respectively) was visually graded according to DIN 4074-5:2008 and the dynamic board MOE was determined (resulting boards of shorter length than log length were only considered to determine the sawing yield, but not for further analysis).

**Table 1: Origin of test material**

Site	Name	Elevation	Stand age [years]	No. of trees	No. of logs	No. of boards*	No. of boards**	
1	Heidenheim	630	104	5	13	71	83	
2	Emmendingen	295	85	5	15	84	96	
3	Rems-Murr-Kreis	480	67	5	14	71	72	
4	Rastatt	393	81	5	14	80	71	
5	Tübingen	469	65	5	12	63	77	
6	Breisgau-Hochschwarzwald	537	55	5	15	90	84	
7	Tuttlingen	980	50	5	11	58	58	
8	Reutlingen	797	58	5	14	78	87	
total					40	108	595	628

\*raw, \*\*edged

**Table 2: Test trees**

Site	n	tree height [m]*	dbh [cm]*
1	5	30.6□4.2	35.0□3.4
2	5	31.9□5.7	35.0□3.3
3	5	29.7□1.1	33.4□4.0
4	5	29.8□3.2	35.0□2.2
5	5	28.4□2.6	34.4□4.3
6	5	30.3□2.2	35.0□4.4
7	5	24.6□1.6	33.8□4.6
8	5	26.5□1.9	35.0□4.0

\*mean ± standard deviation

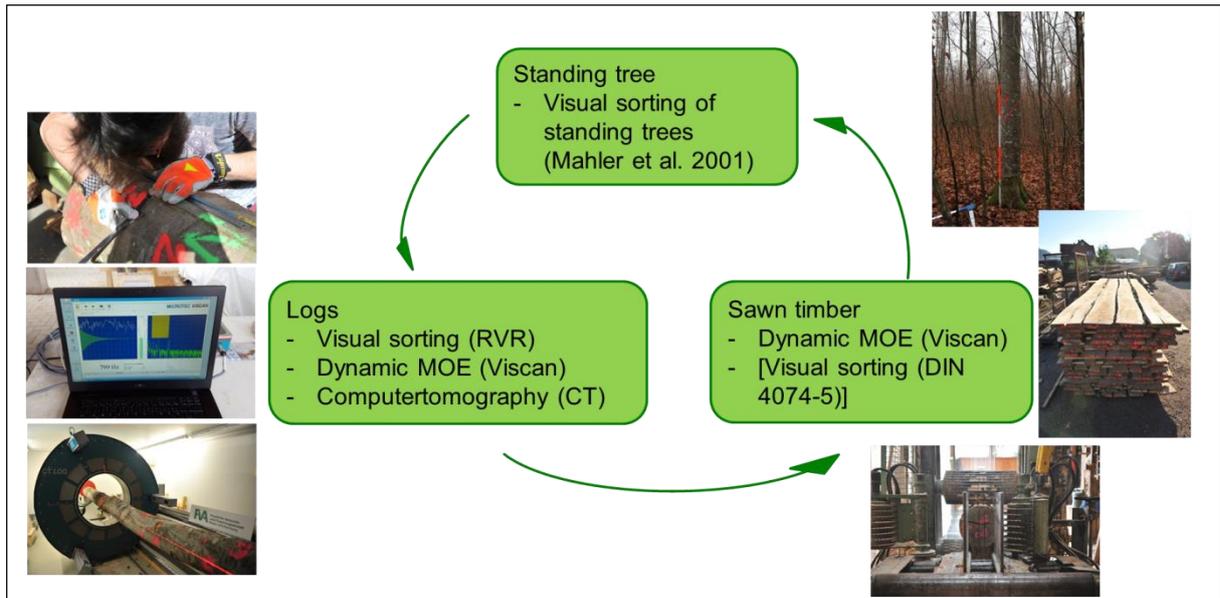
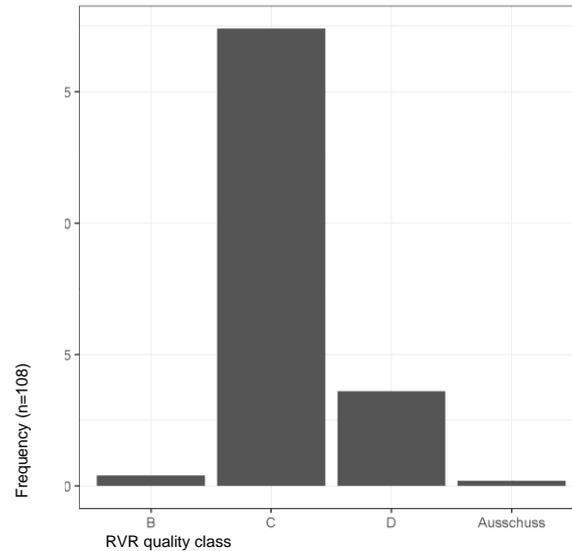


Figure 2: Methods procedure and data obtained in the single steps

## RESULTS

### *Roundwood classification*

Roundwood classification according to RVR (DFWR&DHWR 2015) resulted in more than 95% of the logs classified in the lower two of potentially 4 quality classes: “C” (87 logs, 81%) and “D” (18 logs, 16%), one log as classified “not suitable for sawing”, 2 logs classified as “B” and no class “A” (Fig. 3). The majority of raw logs were classified as “C”-timber of average quality characterized by more frequent or more prominent quality characteristics (DFWR&DHWR 2015). Logs were downgraded mostly due to the size of knots which were either sound and still visible on the stem periphery or only hardly ingrown due to the small stem diameters (size of Chinese moustache). As the dimension of the trees was comparatively small (Table 2) at the time of felling, this classification of the logs was expected in relation to the thresholds given for the RVR grading criteria. The grading thresholds of RVR (DFWR&DHWR 2015) were however agreed with respect to the actual conventional processing strategies for beech wood in the hardwood sawmilling industry reflecting on roundwood of larger dimension (usually top diameter over 40 cm) aiming for sawn timber to be used in visual rather than structural applications.



**Figure 3: Classification of logs to quality classes according to RVR (DFWR&DHWR 2015)**

Breakdown of the 108 logs (37.1 m<sup>3</sup>) gained in 595 raw boards (38.6% yield) and 628 edged, full-length boards (35.8% yield), while approximately 3% of the volume was cut to trimmed boards (length of 2.0-3.8m length for volume yield optimisation, boards were not considered in the following analysis). This volume yield is considerably lower than the average yield reported for softwood breakdown to structural timber (50 – 60%).

### Board characterisation

Visual strength grading according to DIN 4074-5:2008 was performed for strength classification of the boards as machine strength grading of hardwood is not commonly used so far. [DIN 4074-5]-quality classes (LS 7 TS < LS 10 TS < LS 13 TS in increasing strength, TS: graded in dry condition) form the basis for strength classification of glue-laminated timber beams made from beech or as hybrid beech-softwood beams (DIBT, 2019), a potential end product for beech boards.

When all grading criteria were applied about 33% of the boards reached the higher classes LS13 and LS10 which are suitable for use as structural timber (Table 3). This share represents about 31% of the sawn timber volume. However app. 50% of the boards was rejected due to the presence of pith. When pith as criterion was neglected, the proportion of LS13 and LS10 increased to 56% of the boards and only 9% were rejected. In this case app. 53% of the sawn timber volume was classified as suitable for structural purposes. Torno et al. (2013) and van de Kuilen and Torno (2014) reported a volume yield of beech lamellas of 26% for similar investigations when applying DIN 4074-5:2008 and 22% respectively when considering the thresholds for beech glulam.

**Table 3: Full length boards: Quality assessment according to DIN 4074-5:2008**

Sorting	total	LS13		LS10		LS7		reject	
	N	N	%	N	%	N	%	N	%
All sorting criteria due to DIN 4074-5	628	119	18.9	90	14.3	108	17.2	311	49.6
All criteria DIN 4074-5 except pith	628	172	27.4	178	28.3	219	34.9	59	9.4

Dynamic modulus of elasticity varied between 10.2 and 23.3 kN/mm<sup>2</sup> for the single, dried and edged boards (Fig. 4) with a mean of 17.0 kN/mm<sup>2</sup> and standard deviation of 2.4 kN/mm<sup>2</sup>. Statistical analysis of the variation revealed the relation to tree-individual internal stem variation (position of log in the tree), tree age, tree dimension or tree growth related to site conditions.

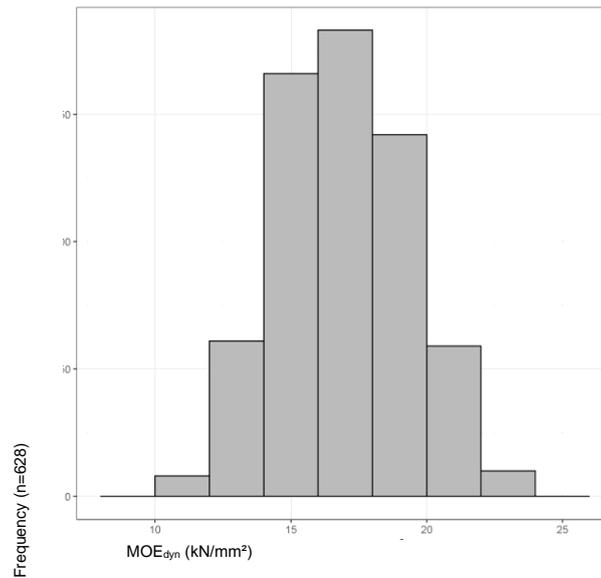


Figure 4: Distribution of MOEdyn of the edged dry boards

Table 4: Dynamic modulus of elasticity MOEdyn (kN/mm<sup>2</sup>) of the edged dry boards

site	age	mean	median	min	max
1	104	17.1	17.3	12.8	23.3
2	85	18.7	19.0	13.2	22.7
3	67	18.2	18.2	13.5	23.3
4	81	16.0	16.0	10.2	20.9
5	65	16.3	16.5	12.0	20.4
6	55	17.6	17.8	13.4	22.1
7	50	14.9	14.7	10.9	20.1
8	58	16.1	15.9	11.6	21.5

Mean MOE<sub>dyn</sub> per site ranges between 14.9 and 18.7 kN/mm<sup>2</sup> (Table 4). Sites from higher altitude (site 7 and 8) show in general a lower MOE<sub>dyn</sub> on the boards than sites from the lower levels (Table 4). Such trends could be considered with respect to a potential pre-selection of sites to identify suitable material for structural purposes.

Arguments for excluding boards with boxed pith from structural grades are the higher likelihood of cracks, splits and deformation, and a lower MOE<sub>dyn</sub> of such boards according to Glos and Lederer (2000). However even though also for the present investigation mean MOE<sub>dyn</sub> of boards containing pith is lower than mean MOE<sub>dyn</sub> of boards without pith, these boards reached an average MOE<sub>dyn</sub> of almost 16 kN/mm<sup>2</sup> (Table 5) which meets the requirements on strength class GL44c and GL 48c for outer and inner lamellas in beech and hybrid glulam (DIBT, 2019).

Table 5: Variation of MOEdyn of the dry edged boards with boxed pith and without pith

	mean	standard deviation	min	max
boxed pith	15.83	2.08	10.9	23.3
no pith	17.98	2.21	10.2	23.3

Standard EN 14080:2013 *Timber structures. Glued laminated timber and glued solid timber. Requirements and National technical type approval Z-0-1-679:2019 for beech glulam and beech-hybrid glulam* set the strength requirements for raw lamellas to be used for beech glulam production. Z-0-1-679:2019 however

also defines a fine-tuning in strength classification proceeding from general DIN 4074:5 sorting classes LS 13 and LS 10 based on additional characterisation with respect to stiffness ( $MOE_{dyn}$ ) and knot size (Table 6).

**Table 6: Requirements to raw lamellas for glued laminated timber. Extract from tables of National technical type approval Z-0-1-679:2019 for beech glulam (DIBt 2019).**

strength grade <sup>a</sup>	GL 28h	GL 32c	GL 36c	GL 40c	GL 44c	GL 48c
Requirements for outer lamellas						
Grade <sup>c</sup>	LS10	LS13	LS13+A <sup>c</sup>	LS13+E14 <sup>d</sup>	LS13+E15 <sup>d</sup>	LS13+A+E15 <sup>e</sup>
$E_{dyn}$	-	-	-	>14	>15	>15
Requirements for inner lamellas						
Grade	LS10	LS10	LS10	LS10+E13	LS10+E14	LS10+E14
$E_{dyn}$	-	-	-	>13	>14	>14

<sup>a</sup> according to EN 14080:2019, <sup>b</sup> according to DIN 4074-5:2019;

<sup>c</sup> knot coefficient < 0,04; <sup>d</sup>  $MOE_{dyn}$  of board > defined  $E_{dyn}$

Table 7 summarises the classification of boards according to the requirements for glued timber structures (homogenous or hybrid beech glulam). Considering all visual criteria of DIN 4074-5 about 1/3 of the boards meets the requirements for glulam, 2/3 fails due to DIN 4074 classification as LS7 or reject. The share would increase to over 50% in case the presence of pith would be excluded from the assessment. If only the mechanical stiffness  $MOE_{dyn}$  would be considered, 493 boards or 79% would be classified as suitable with respect to mechanical properties.

**Table 7: Classification of edged boards according to Z-0-1-679:2019**

Strength grade	GL28h	GL32c	GL36c	GL40c	GL44c	GL48c	reject
All criteria	90	3	1	3	51	61	419
All criteria except pith	178	8	1	9	87	67	278

## CONCLUSION

Due to the selected range of log dimension in the smaller size classes (log mid diameter without bark from 17 to 38 cm, median 28 cm) a high share of the sawn boards contained pith which resulted in a large proportion of rejected boards in strength grading according to the relevant DIN 4074-5:2008. The same problem was also reported by Torno et al. (2013), van de Kuilen and Torno (2014) and Schlotzhauer et al. (2019). Nevertheless, also boards visually rejected due to the presence of pith can reach levels of mechanical stiffness  $MOE_{dyn}$  which meet the requirements for beech glulam for strength class GL 40 and higher. In the light of the large yield losses due to the presence pith in boards from small-diameter hardwood logs, Schlotzhauer et al. (2019) and Kovryga et al. (2019) discuss options for an adopted sorting and grading of hardwood for structural grades based on mechanical characterisation.

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## Discolouration of oak wood caused by different light sources

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**Keywords:** Oak wood, discolouration, lighting, LED

### ABSTRACT

Discolouration is recognised to be the first sign of wood photo-degradation, as colour changes are more pronounced than other indicators during the initial phase of irradiation. In addition, discolouration can be a significant aesthetic concern, especially for wooden interior objects even if no other effects of degradation are noticeable. The two main kinds of light sources to which indoor objects can be exposed are solar radiation and artificial lighting. LEDs are becoming the dominant artificial light type in both private and public spaces. However, although LEDs have lower output in the UV range, they emit more in the blue light range, which could influence wood discolouration. The present study is a part of the research aimed at evaluating colour changes and providing discolouration models of different wood species depending on the light type. Oak (*Quercus robur*) wood was used for representing the lower limits of expectable discolouration imparted by light to wood. Fluorescent UVA-340 and UVA-351 type lamps were used for representing UV of direct sunlight and UV of sun through window glass, respectively. The artificial lighting sources used in the study were incandescent lamps, fluorescent lamps and LED lamps with colour temperature of 3000 K and 6000K. Wood discoloration was monitored by spectrophotometric measurements of reflectance spectra and chromaticity parameter calculations using CIELAB colour system. The results showed that exposure of oak wood to different artificial light sources of similar dose (lux hours) resulted in discolouration, which differed both in the total extent (DEab) as well as in the changes of the colour parameters. Although all used light sources caused wood darkening, the changes in the chromaticity parameters a\* and b\* differed. The results demonstrated that greater total discolouration was caused by the light sources emitting more in the UV range. Similar discolouration was observed for equal UV radiation doses (Wh/m<sup>2</sup>) caused by exposure to the fluorescent UVA type lamps of different spectral composition.

### INTRODUCTION

Wood colour is one of the elements imparting aesthetic value to wooden objects. The colour of any surface depends on its chemical composition, namely the chromophores which absorb definite wavelength of the visible light. Consequently, discolouration is related to some changes in the composition of these groups. When biotic factors are excluded, wood discolouration is mostly the result of alteration in wood chromophores caused by thermal oxidation or photo-induced chemical reactions. The thermal oxidation reactions in wood are of quite low rate under normal conditions (Matsuo et al. 2011). In contrast, photo-degradation rate is found to be quite rapid process at short exposure times with becoming slower in further exposure (Wang and Ren 2008, Timar et al. 2016). Thus, photo-degradation is the dominant cause of wood colour change indoors. In addition, it has been found that the colour change caused by irradiation can be more pronounced than the changes in wood chemical composition (Tolvaj and Mitsui 2005). Wood discolouration is mostly associated with chemical transformation in lignin with formation of quinones and quinone like structures with some absorption maximums in the visible light range 400 – 500 nm (Tolvaj and Faix 1995, Wang and Ren 2008). Apart from lignin, also extractives can play certain role in wood photodegradation (Zahri et al. 2007, Chang et al. 2010).

Discolouration of wood due to exposure to irradiation is an inherent wood property. The extent of it depends mostly on the wood species, the spectral composition of the incident light, as well as on the radiation dose which is a product of radiation intensity and exposure time (Tolvaj and Mitsui 2005, Kataoka et al. 2007,

Oltean et al. 2008, Timar et al. 2016). Another factors that has certain effect on wood photo-discolouration is temperature. Concerning indoor environment, it usually is of secondary importance because the variations are too small to exert significant influence (Mitsui and Tsuchikawa 2005).

Exposure to UV radiation is well recognized to be the main cause of photo-degradation of wood. However, the region of the visible light of the shorter wavelength is found to induce chemical transformations in wood as well. Moreover, the discolouration pattern can differ depending on the wavelength range included in the incident radiation (Kataoka et al. 2007, Živković et al. 2014, Cirule et al. 2016).

There is a great number of publications analysing different aspects of wood photo-degradation and discolouration. The solar radiation or its artificial imitation with mimicking the whole spectrum or only the UV portion are the dominant radiation sources employed for analysing different aspects of wood photo-discolouration. However, there is a lack of knowledge regarding the potential influence of artificial lighting on wood discolouration. On the other hand, information on the material sensitivity to different light sources can help to elaborate the most optimal exposure strategy.

The types of the most widely used sources of artificial light have changed considerably over the last decades. The incandescent lamps that were the most common artificial light source for a very long period were phased-out in the EU because of the very high energy losses. They were substituted by different kinds of fluorescent lamps with considerably improved efficacy (lumens per watt). From an environmental point of view, the fluorescent lamps have significant drawback due to containing mercury. Today LED lamps are becoming the dominant light source for different indoor environments due to their superior efficacy and long lifetime. Although the LED lamps emit hardly any radiation in the UV range, it is typical for them to emanate quite a lot of visible light of the shorter wavelength, which can cause wood photo-degradation.

The present study is a part of the research aimed at evaluating colour changes and providing discolouration models of different wood species depending on the light type. In the preliminary experiments, oak (*Quercus robur*) wood was found to be the most resistant to discolouration among other species widespread in the Northern Europe which agrees with the results observed by Oltean et al. (2008). Consequently, oak wood is used in the research for representing the lower limits of expectable discolouration.

## EXPERIMENTAL METHODS

In this study, 5 specimens each from different oak board (*Quercus robur*) were exposed to each of the light sources. The UVA-340 and UVA-351 type fluorescent lamps were used in the weathering chamber (Q-Lab USA) for simulation of the UV radiation of the direct sunlight and sunlight through window glass, respectively. The irradiation intensity was  $980 \mu\text{W}/\text{cm}^2$  for both type of lamps. The artificial light sources used were: incandescent lamps (60 W), fluorescent lamps, and LED lamps of two colour temperatures, namely 3000 K and 6500 K. The spectral composition of the artificial light sources is provided in Fig. 1.

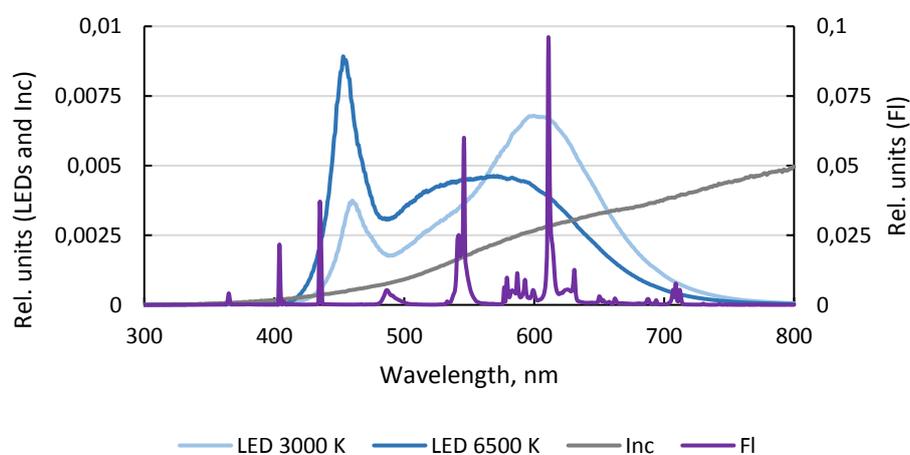


Figure 1: Spectral composition of the light emitted by incandescent (Inc), fluorescent (Fl) and LED lamps

The exposure to the artificial light was performed in chambers with all inside walls coloured black. The light intensity on the surface of specimens were as follow: 1150 lx for incandescent lamps, 1700 lx for the fluorescent lamps, and 6000 lx for both kind of the LED lamps.

A spectrophotometer Minolta CM-2500d (standard illuminant D65, d/8° measuring geometry, 10° standard observer, measuring area Ø 8 mm) was used for measuring of reflectance spectra and colour parameters. Colour was expressed according to the CIELAB colour model (*Commission Internationale de l'Eclairage, 1976*) as colour parameters L\*, a\*, b\*. The total colour change DEab was calculated from the colour parameter differences between the initial and resulting values DL\*, Da\*, Db\* according to the formula:

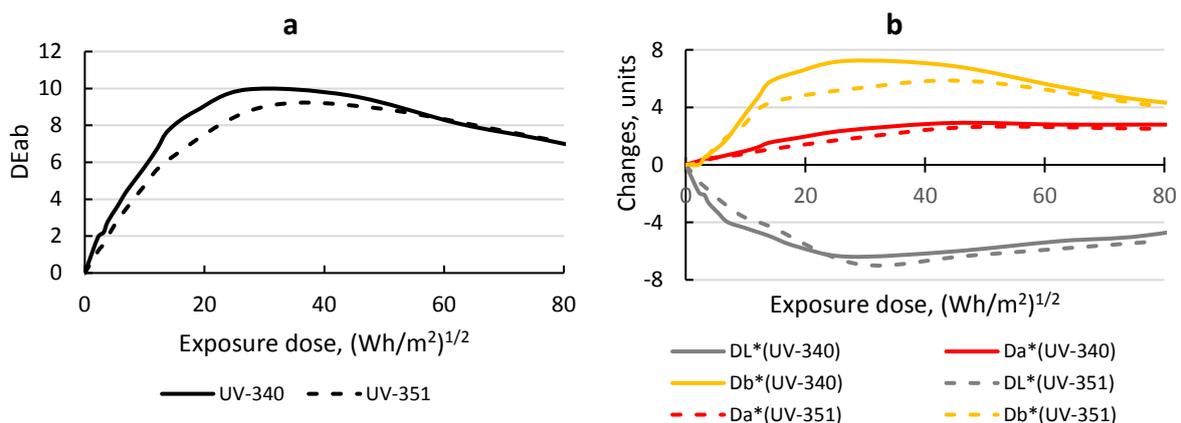
$$DEab = (DL^*^2 + Da^*^2 + Db^*^2)^{1/2} \quad (1)$$

Spectrophotometrical measurements were performed before and at definite exposure intervals always at the same five points on the surface of each specimen.

## RESULTS AND DISCUSSION

In a preliminary experiment, in which specimens were exposed to the sunlight in three ways, namely without any protection, under window glass and under glass transmitting only the light of wavelengths longer than 390 nm, it was detected that the sunlight with the UV radiation filtered off had caused hardly any discolouration (DEab of 0.4 units) of the oak wood after 20 h of exposure. In its turn, colour changes recorded after the same time-span for the specimens exposed to the direct sunlight and for those covered with a window glass were 3.9 and 1.9 DEab units, respectively. These results clearly demonstrated that in the case of both direct sunlight and sunlight through window glass oak wood discolouration is predominantly caused by UV radiation. Artificial weathering tests were performed for evaluating potential discolouration of oak wood caused by exposure to the direct solar irradiation as well as to solar radiation through window glass. According to the weathering chamber producer (Q-Lab), the fluorescent lamps of UVA-340 and UVA-351 types used for wood irradiation in the present study differ by the emitted radiation. The most relevant difference is a lack of some radiation at the shorter wavelength range for the UVA-351 lamps compared with UV-340 lamps.

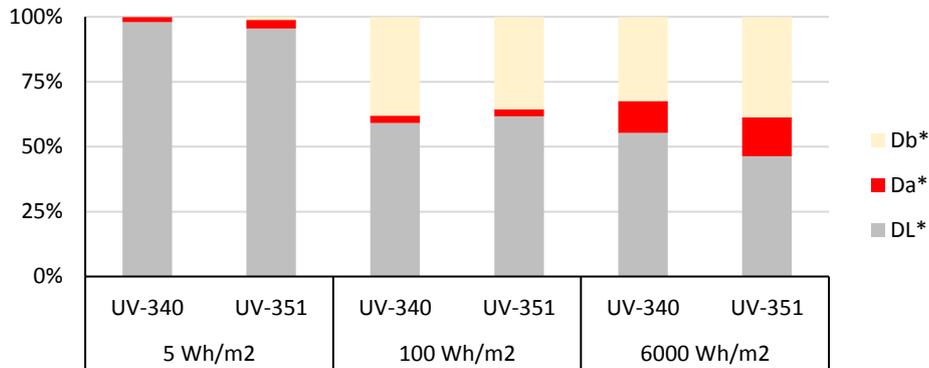
The colour change results from artificial weathering tests show that there are no substantial differences neither between the total discolouration values (Fig. 2-a) nor between the alteration pattern of colour parameters (Fig. 2-b) caused by UVA-340 and UVA-351 fluorescent lamps when the effects of equal radiation dosage (W h/m<sup>2</sup>) are compared.



**Figure 2: Discolouration (a) and alteration in colour parameters (b) of oak wood caused by UVA-340 and UVA-351 type fluorescent lamps depending on the exposure dose**

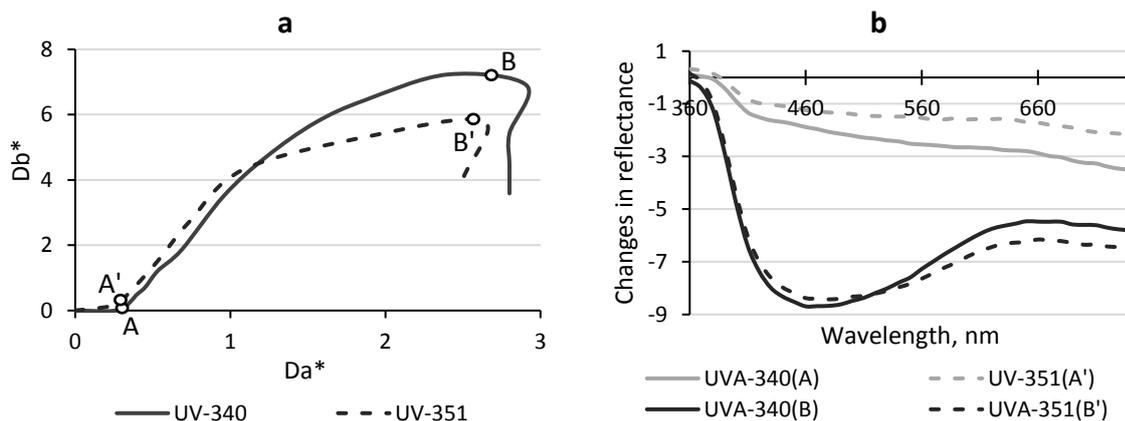
The similarity of the trends regarding the changes in the colour parameters implies that the differences in the radiation spectral composition do not substantially influence the photo-discolouration reactions of oak wood. The general discolouration trend due to the UV irradiation includes wood darkening (negative DL\*)

values) and increase in chromaticity parameters (positive  $Da^*$  and  $Db^*$  values) which in the CIELAB colour space corresponds to reddening and yellowing, respectively. Similar discolouration pattern has been found as a typical for a number of wood species exposed to UV irradiation (Deka and Petrič 2008, Oltean et al. 2008). However, except for the very initial period of exposure when changes in wood lightness  $L^*$  are the dominant contributor, both darkening and yellowing with more or less equal input are prevailing in oak wood discolouration (Fig. 3).



**Figure 3: Contribution of each colour parameter to the total discolouration of oak wood caused by exposure to UVA-340 and UVA-351 type fluorescent lamps for definite exposure doses**

Oltean et al. (2008) have reported similar results regarding dominance of  $L^*$  and  $b^*$  in discolouration of oak wood due to simulated indoor sunlight exposure. Zahri et al. (2007) have found that extractives, which are quite abundantly present in oak wood, can significantly contribute to oak wood yellowing due to UV irradiation. In the present study, as it can be seen from the graph, in which the changes in the chromaticity parameters  $a^*$  and  $b^*$  are plotted (Fig. 4-a), the discolouration commences with increase in redness ( $a^*$ ) that after reaching the maximum value stays more or less constant. On the contrary, some induction time was detected for the onset of yellowing and a decrease in yellowness values was recorded after passing the maximum.



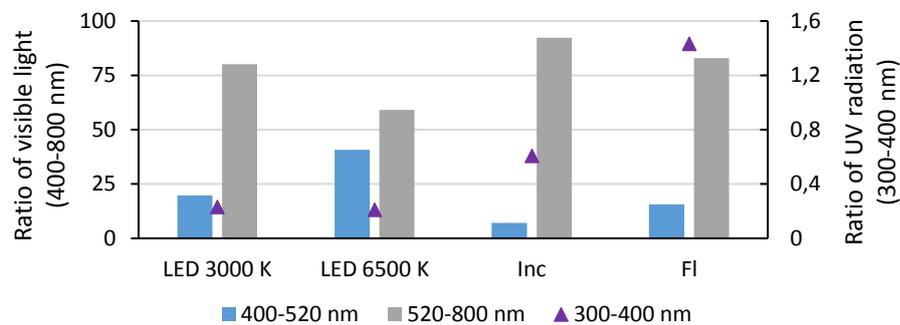
**Figure 4: Relation between changes in chromaticity parameters (a) and reflectance difference spectra (b) of oak wood caused by exposure to UVA-340 and UVA-351 type fluorescent lamps**

In Fig. 4-b, the reflectance difference spectra of the specimens are provided for the exposure times corresponding to the chromaticity changes at the points A and A' as well as B and B' in Fig. 4-a. The discolouration with hardly any changes in  $b^*$  (points A, A') corresponds to a progressive decrease in reflectance towards the longer wavelength. On the other hand, the discolouration caused predominantly by the yellowing (points B, B') corresponds to a reflectance difference spectra with a pronounced band of a

decrease in a region around 460 nm which is typically associated with formation of quinone structures due to lignin photo-degradation (Pandey and Vuorinen 2008).

In the above mentioned preliminary experiment, the recorded UV radiation intensity ( $\text{W}/\text{m}^2$ ) for the sunlight through the window glass was ca.  $45 \pm 5 \%$  from that of the direct sunlight. Consequently, this observation combined with the results of wood exposure to the UV in the weathering chamber suggests two times slower oak wood discolouration caused by its exposure to the solar irradiation through the window glass compared with that caused by the direct sunlight. However, it should be noted that in the preliminary experiment the specimens were exposed to irradiation and accordingly the radiation intensity was recorded only on clear days during the hours when the UV intensity of the direct solar radiation was at least  $10 \text{ W}/\text{m}^2$ . Other ratio of the UV radiation could be transmitted through the window glass in different conditions. Thus, the suggestion about the comparative rates of oak wood discolouration caused by exposure to solar radiation is more accurate for the solar radiation of higher UV intensities.

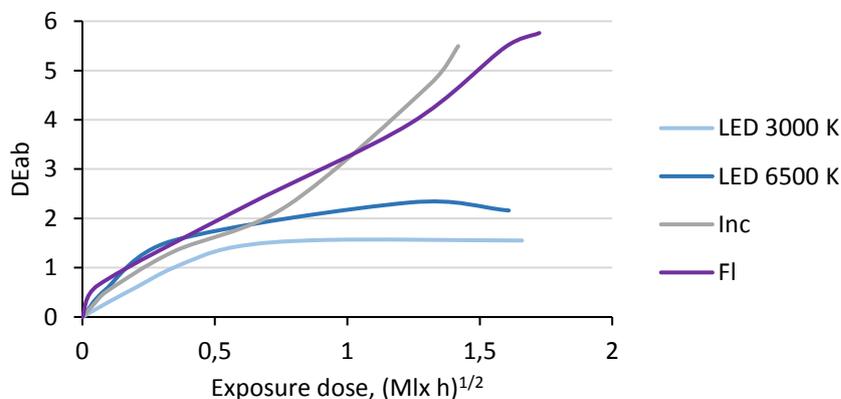
In Fig. 5. for characterization of the spectral composition of the lamps, the radiation emitted by different light sources in the wavelength range 300 – 800 nm is split into three regions considering their potential effect on wood.



**Figure 5: Ratio of different wavelength ranges in the radiation emitted by the LED, incandescent (Inc), and fluorescent (Fl) lamps**

It can be seen, that, although the ratio of the emittance in the UV range is quite low for all lamps, yet the incandescent lamps and especially the fluorescent lamps emit considerably more in the UV range compared to the LED lamps. For their part, the LED lamps, and especially those of higher colour temperature, irradiate more in the visible light region (400 – 520 nm) that has been identified to cause photo-degradation of wood (Kataoka et al. 2007, Živković et al. 2014, Cirule et al. 2016).

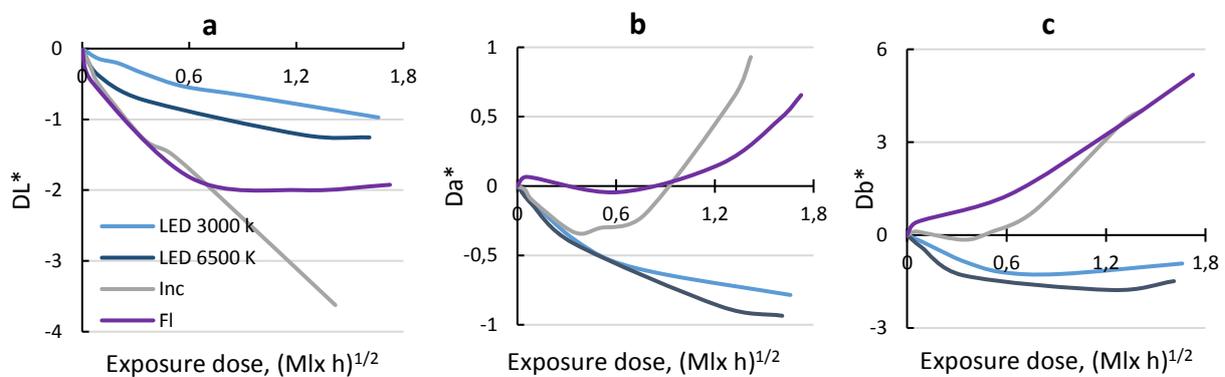
The progress of the total colour changes caused by different artificial light sources depending on the exposure dose is presented in Fig. 6. It should be noted that the experiments are still ongoing and the results presented here characterize the discolouration of oak wood exposed to illumination for ca. 6 000 hours with light intensity of 500 lux or 15 000 hours with 200 lux. Quite fast discolouration signalling of the photo-induced reactions was detected in the initial period for the specimens exposed to all lamps with the most rapid colour change induced by the fluorescent lamps.



**Figure 6: Discolouration of oak caused by exposure to the LED, incandescent (Inc) and fluorescent (Fl) lamps depending on the exposure dose**

However, it is suggested that colour difference should be above two DEab units to be perceptible for human eye, implying that visually the surface remains unchanged before reaching this value (Mokrzycki and Tatol 2011). The lowest exposure dose for reaching discolouration of the visually perceptible level ( $DE_{ab} > 2$ ) was also provided by the fluorescent lamps which well agrees with the highest ratio of UV radiation in the spectral composition of these lamps. Quite similar pattern of colour changes was observed for both fluorescent and incandescent lamps with no maximum reached at the moment of summarising these results. However, somewhat slower discolouration in the initial period and faster in the later course was detected for the specimens exposed to the incandescent lamps. In contrast, exposure to the LED lamps caused hardly any visually perceptible discolouration, although spectrophotometrically were detected some colour changes. These results suggest that concerning discolouration LED lamps regardless of the colour temperature are safer for oak wood than incandescent and fluorescent lamps.

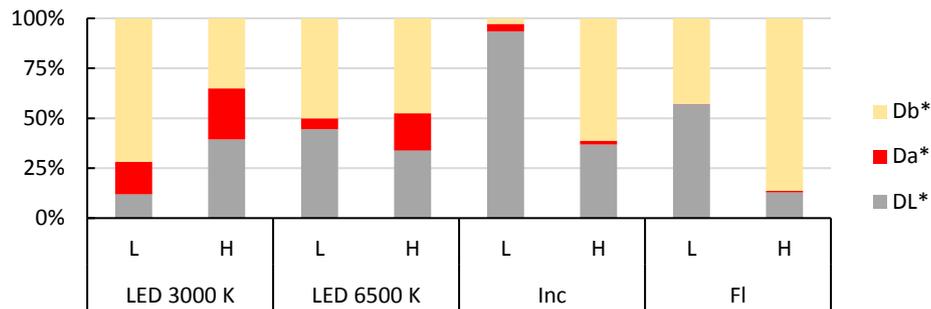
Analyses of the changes in the colour parameters  $L^*$ ,  $a^*$ ,  $b^*$  show considerable differences in the patterns of changes depending on the light source used for the exposure (Fig. 7). It indicates that the resultant colour of oak wood developed due to exposure to artificial light sources can differ in the colour tone depending on the light source even when the total discolouration extent measured in  $DE_{ab}$  units is similar.



**Figure 7:** Changes in colour parameters of oak wood caused by different type of light sources depending on the exposure dose: a) changes in lightness ( $DL^*$ ), b) changes in redness ( $Da^*$ ), c) changes in yellowness ( $Db^*$ )

Regarding the changes in the lightness ( $DL^*$ ), all employed lamps caused darkening of wood for the analysed exposure doses (Fig. 7-a). In contrast to other light sources, for which the darkening progressed with accumulation of the exposure dose, in the case of fluorescent lamps a plateau was reached quite fast. More substantial differences were found for patterns of changes in chromaticity for different kinds of light sources. Similar trends were observed for the LED lamps regardless of the colour temperature with quite stable decrease in both yellowness as well as redness and only slightly more changes in yellowness. In contrast, increase in yellowness throughout the course of discolouration was detected for the specimens exposed to the other two types of lamps. However, definite exposure dose was required for initiation of the yellowing process in case of the incandescent lamps while it started quite rapidly for the specimens exposed to the fluorescent lamps (Fig. 7-c). Regarding the changes in the redness (Fig. 7-c), the fluorescent and incandescent lamps exerted different effect on oak wood. Considerable fluctuation of the changes in the redness was detected for the specimens exposed to the incandescent lamps. Initial decrease was followed by increase for higher exposure doses implying that the products of the primary photo-induced reactions were involved in the further chemical transformations. In case of the fluorescent lamps, quite high exposure doses in the initial part of irradiation caused hardly any changes in the redness while an increase in this colour parameter was observed due to the further exposure.

Apart from the differences between patterns, the contribution of the changes in each colour parameter to the total colour change differs depending on the light source. Moreover, the contributions vary with the progress in exposure. In Fig. 8, the contribution of changes in each colour parameter to the total discolouration is given for low (L) and high (H) exposure doses, ca. 2.6 and 0.6  $Mlx\ h$ , respectively.



**Figure 8: Contribution of each colour parameter to the total discolouration of oak wood caused by exposure to LED lamps, incandescent lamps (Inc), and fluorescent lamps (Fl) for high (H) and low(L) exposure doses**

It can be seen that the total changes in the chromaticity parameters (Da\* and Db\*) have the dominant effect in the discolouration caused by the LED lamps with increase in effect of redness changes for the higher dose. For both the incandescent and fluorescent lamps, dominance of chromaticity changes was only observed for the higher exposure dose. The discolouration caused by low dose exposure was mainly due to the changes in wood lightness, especially in the case of incandescent lamps when more than 90 % of the discolouration can be attributed to the wood darkening. In contrast to discolouration caused by the LED lamps, hardly any contribution of the changes in the redness was found to be typical for the exposure to the incandescent and fluorescent lamps regardless of the exposure dose. Both the dominance of the changes in the lightness at the onset of discolouration as well as the low contribution of the changes in redness agree with the results detected for the discolouration caused by exposure of the specimens to UV radiation. It implies that, although the ratio of the UV radiation in the light emitted by these lamps is not high, the UV-induced reactions prevail in altering oak wood colour when it is exposed to light sources emitting UV radiation even at low doses.

## CONCLUSIONS

1. The differences in spectral composition of UV radiation of the direct sunlight and the sunlight filtered through the window glass modelled by UVA type fluorescent lamps do not have any substantial effect on oak wood discolouration.
2. The rate of oak wood discolouration as well as the pattern of changes in the colour parameters L\*, a\*, b\* differ depending on the type of the artificial light source used for illumination.
3. The LED lamps regardless of colour temperature cause substantially less discolouration of oak wood in comparison with the incandescent and fluorescent lamps, at least for the exposure doses up to 3 Mlx h.
4. The similarity in discolouration pattern with that caused by exposure to UV radiation implies that the UV-induced reactions prevail in altering oak wood colour when it is exposed to the incandescent and fluorescent lamps.

## ACKNOWLEDGMENTS

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## Seasonal changes of uprooting safety factor of different tree species

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**Keywords:** Uprooting safety, seasonal effect, wind load

### ABSTRACT

In this study we focused on the seasonal effect on root stability in winter and summer time. Measurements were carried out at the botanical garden of University of Sopron. The presence of leaves in summertime may increase the wind load due to increasing the crown surface area hit by the wind. In wintertime the crown surface area of the broad-leaved trees is reduced, while the aerodynamic drag coefficient is increased. These two effects may compensation each other. We carried out experimental measurements to clarify the seasonal variation of wind load and uprooting safety factors (SF) of different tree species.

### INTRODUCTION

Tree root mechanical stability is important in urban environment and in forest stands. The stability of urban trees is a key question that affects everyone. Diseased and unstable urban trees pose much risk for everyone, and are a serious liability for municipalities in case of an accident. Tree stability assessment is therefore of the utmost importance. In the meantime, it tends to be much neglected in many areas.

The main load causing tree failure in urban areas is the wind load. This can be simulated during static pulling test and used during the “dynamic pulling test” or dynamic tests. The wind load may be changing as well, according to the foliage, which was the focus of this investigation.

The pulling test, while well established and accepted, has several drawbacks, both in terms of reliability and ease of use. Dynamic root stability determination is based on real life wind loads, and is therefore more appropriate for assessing real life risks. It is also much simpler to carry out than the traditional pulling test, with the only drawback being that it requires windy weather to carry out.

The pulling test is based on affixing a cable at approximately mid-height on the tree to be evaluated, and applying a moderate load, while measuring the inclination at the base of the trunk. The induced inclination is very moderate (less than 0.2 degrees), to make sure that the test itself does not damage or start uprooting the tree. Based on the measured load, it is possible to extrapolate to the approximate torque required to uproot the tree. The maximum torque that may arise due to adverse weather conditions can also be calculated based on the crown surface, aerodynamic drag factor and the maximum wind velocity likely to occur in the area. The ratio of the two values is the safety factor (SF) of the given tree. If this value is above 1.5, the tree is declared safe, while a SF below 1 signals high risk. In-between these two values, the safety of the tree is considered uncertain.

The advantage of the pulling test is that it is a fairly straightforward, well-researched and established method. On the other hand, the applied static load approximates the real life situation, where trees are subjected to dynamic loading, rather poorly. It is also quite cumbersome; requires heavy equipment (high capacity cable and ratchet) and a ladder to be carried to the test site, lengthy preparation and physical exertion during testing. The pulling cable has to be fastened to some well-secured object on the ground (like another tree trunk or stump) that may or may not be readily available.

During the so-called “dynamic” pulling tests the wind, and especially the wind gusts should be measured (while measuring the inclination of the trunk similar to the static pulling test is also necessary). A high sampling-rate anemometer on a 10 m height pole is great solution for this task.

If there is enough wind to lean the trunk but not uproot or break the trunk, the inclination data can be collected, and safety factor can be estimated similarly to the extrapolation for pulling test. Our goal was to examine if the presence or lack of foliage affect the dynamic safety factor.

## MEASUREMENTS

Dynamic loading was used in this study. For the measurements 3 species (*Robinia*, *Fraxinus*, *Platanus*) were selected in the botanical garden at University of Sopron. Different trees of these species were estimated by DynaRoot system in winter and summer (Fig 1).

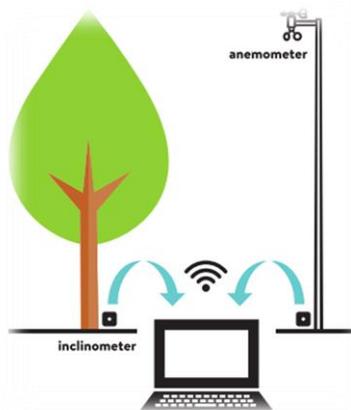


**Figure 1: *Robinia pseudoacacia* with and without leaves in summer and early spring**

Dynaroot system has 3 parts, anemometer, inclinometer and evaluation software. Anemometer is an instrument for measuring wind velocity at or near the tree to be evaluated. The closer the anemometer is to the tree the better, but, depending on wind velocity DynaRoot may provide reliable data even with measurements taken several kilometers away. Ideally the anemometer should be clear of buildings or other objects that may obstruct the wind, at a height of at least 10 m (Fig 2).

Inclinometer is able to record inclination of tree's movement during wind blow. The sampling frequency for inclination was 10 Hz. Inclination data is also recorded on a data card along with the exact time of measurement (Fig 2).

Evaluation software: a PC software for evaluating wind velocity, x and y inclination. The data, recorded over periods of several hours (varies depending on weather conditions), are transferred from the anemometer and inclinometer on memory cards. Both wind velocity and inclination should be recorded at the same time (Fig 2).



**Figure 2: Schematic of the DynaRoot system (Buza and Divós 2016)**

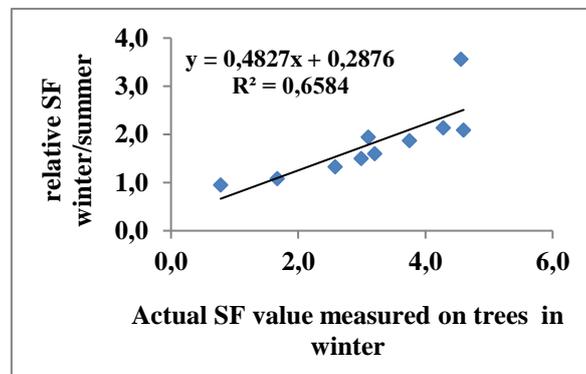
## RESULTS

Significant difference in safety factor was examined. Generally, the safety factor measured during winter was higher than the ones measured during summer. It seems that the presence of foliage increases wind load. The only exception was examining the *Fraxinus* trees in which case the difference was very small. Table 1 shows the ratios of safety factors measured during winter and summer.

**Table 1: Preliminary results of the safety factor ratio, winter/summer**

Species	SF <sub>winter</sub> /SF <sub>summer</sub>	Species	SF <sub>winter</sub> /SF <sub>summer</sub>
<i>RobiniaIpseudoacacia</i>	1.5	<i>Platanus occidentalis</i>	1.6
<i>RobiniaIpseudoacacia</i>	2.1	<i>Platanus occidentalis</i>	1.9
<i>RobiniaIpseudoacacia</i>	2.1	<i>Platanus occidentalis</i>	1.3
<i>Fraxinus excelsior</i>	1.0	<i>Platanus occidentalis</i>	3.6
<i>Fraxinus excelsior</i>	1.1	<i>Platanus occidentalis</i>	1.9

Actual safety factor which were measured in 2 different seasons were compared with relative safety factor (winter/summer). As you see in (Fig 3) results show as expected there is acceptable correlation ( $R^2 > 0.60$ ) between relative SF<sub>(winter/summer)</sub> and SF in winter. Similar correlation was not found as comparing SF<sub>(winter/summer)</sub> and SF in summer.



**Figure 3- comparison between relative SF (winter/summer) with actual SF in winter**

The broad-leaved tree safety factor is significantly higher in wintertime, while conifer safety factors are rather similar at winter and summertime. This indicates that wind load on broad-leaved trees in the summertime is more complicate than in wintertime (Fathi et al 2019).

With this simple comparison could get that information about broadleaves, during summertime their construction needs more detailed research on each effect.

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## Structured wood surfaces for flooring – Coatings to obtain high resistance and natural look and feel

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**Keywords:** flooring, coating, structure, brushing, resistance

### ABSTRACT

The aim of the project Surf~Parquet was the improvement of the durability against pollution and chemical influences while sustaining the natural look and the haptic features of the structured wood surface of floors. Coatings were used to improve the resistance of the surfaces, weak spots in the coatings were analysed by microscopy and based on this application techniques were improved. In the project, several variants of structured wood flooring with uniform matt coating films over the topography were obtained, which resulted in a natural look and feel as well as high resistance.

### INTRODUCTION

Surface structuring of wood flooring such as brushing is a means of emphasising the natural structure of wood and giving it a natural look and feel. Coating these wood surfaces with deep structures is difficult because state of the art coatings either obscure the wood structure with a relatively thick film or they are not continuous. The aim of the project Surf~Parquet was the improvement of the durability against pollution and chemical influences while sustaining the natural look and the haptic features of the structured wood surface of floors.

### EXPERIMENTAL METHODS

A selection of 76 combinations of coated wood floorings was defined in collaboration with the project's SME user committee and included:

- uncoated floors:
  - top layers of oak (O), larch (L) and maple (M)
  - various pre-treatment variants (sanded (S), medium brushed (MB), dense brushed (DB))
- coating materials in 25 different coating variants:
  - wax/oil-wax (Wax/oil-wax)
  - UV-oils (UV\_oil)
  - oxidative curing oils (Oxy\_oil)
  - UV lacquers (UV\_lacquer)
  - water-based lacquers (wb\_lacquer)

The dry film thickness was determined on cross-section specimens of the sanded variants under the microscope. The categorization into film-forming systems (dry film thickness > 20 µm) and non-film-forming systems (dry film thickness < 20 µm) was carried out on the basis of these measurements in order to assign the coating systems to the different requirement groups. Coated flooring samples were characterized in terms of colour, gloss and appearance and tested with 10 % ammonia in a screening of resistance. The visual impression of coated and uncoated surfaces was determined using colour scan, colour and gloss measurements of coated and uncoated boards. Variants which passed the ammonia screening in, i.e. degree ≥ 4 for non-film-forming or degree = 5 for film-forming coatings, were used for resistance tests according to EN 12720 with further chemicals. These test chemicals were acetic acid, cleaning agent, coffee, ethanol, paraffin oil and water in order to check the chemical resistance against typical liquids to be

found in every household. For all liquids one concentration with two or three different exposure times were chosen. 3D topography of structured surfaces of oak, larch and maple was studied with SEM and light microscope. Coatings were used to improve the resistance of the surfaces, weak spots in the coatings were analysed by microscopy and based on this application techniques were improved.

A test-person study was conducted with 30 persons at IHD and 30 persons at HFA involving only non-expert persons equally distributed over gender and age groups 80 samples were evaluated by every person.

## RESULTS AND DISCUSSION

Most coating systems on maple caused only a slight colour change compared to uncoated surfaces, in terms of brightness, the green-red axis and the blue-yellow axis of the CIELab colour space. The coating systems on oak and larch tended to cause the colour to shift towards dark and red and yellow. However, an intensified impression of the wood on the coated surface compared to the uncoated surface was not necessarily seen negatively as unnatural appearance (Fig. 1).



*Figure 1: Scans of uncoated and coated wooden floors with maple, larch and oak top layer*

The coating systems showed in general a low gloss level (Fig. 3), which resembled the natural appearance of the wood surface. Individual systems showed a slightly higher gloss, which in absolute terms was still in the matt sensation range. Even in the coated samples, the gloss across the fibre was lower than along the fibre.

Weak spots in the structured coated surfaces were studied on Oak with the SEM (Fig. 4) and subsequently avoided by improved coating techniques. Finally, good practice examples for film formation were found (Fig. 5). Although the topography of the surface structured by brushing had deep valleys and narrow peaks (compared to sanded wooden surfaces) the film formed a uniform and continuous layer without weak spots on top of the surface.

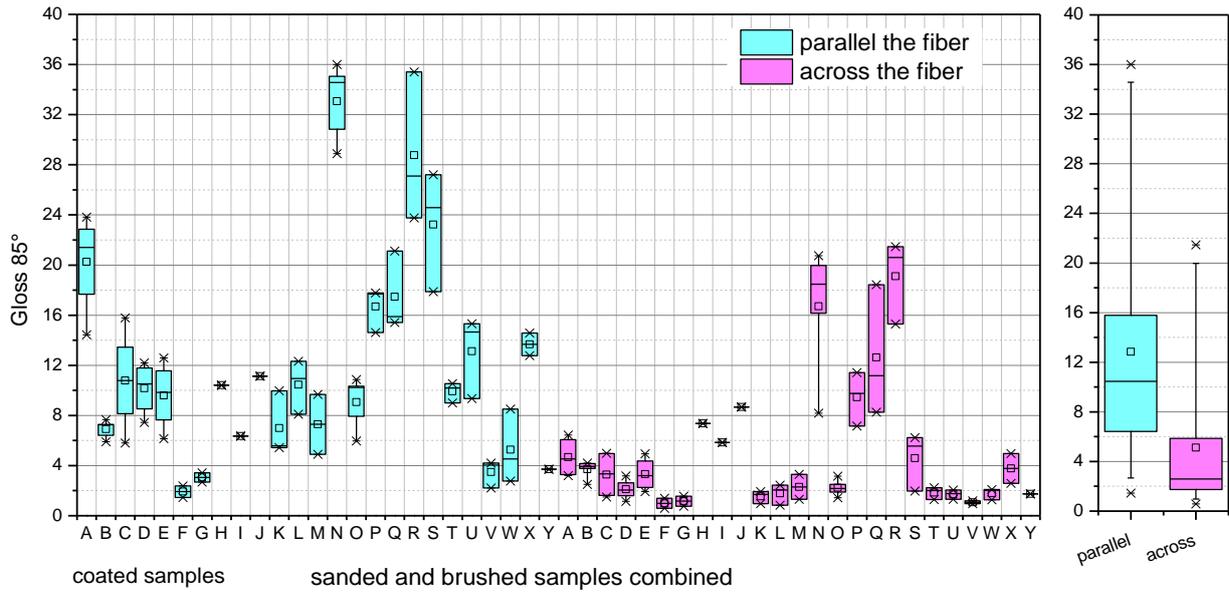


Figure 2: Gloss measurement of coated wood floors - comparison of coating systems A to Y

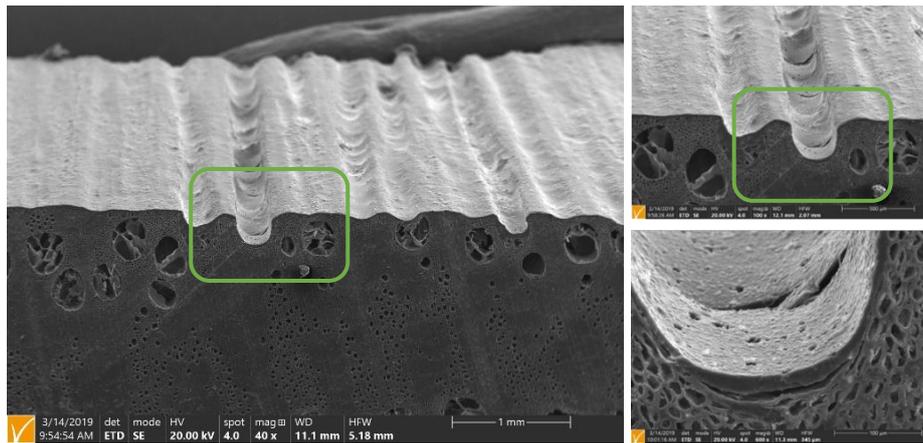


Figure 3: Weak spot analysis with SEM-images of medium brushed oak coated with UV-Oil L – from overview into the detail – coating cracks in a vessel

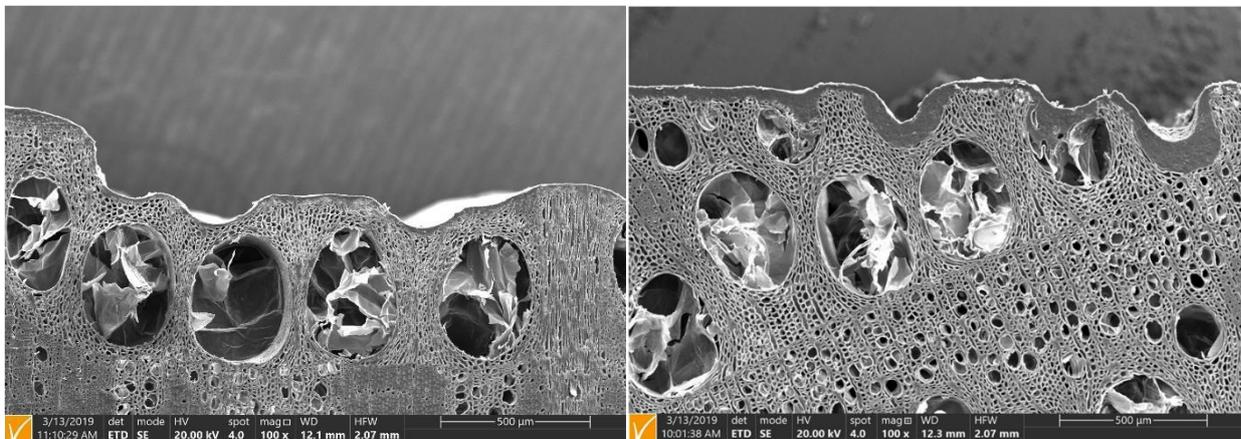
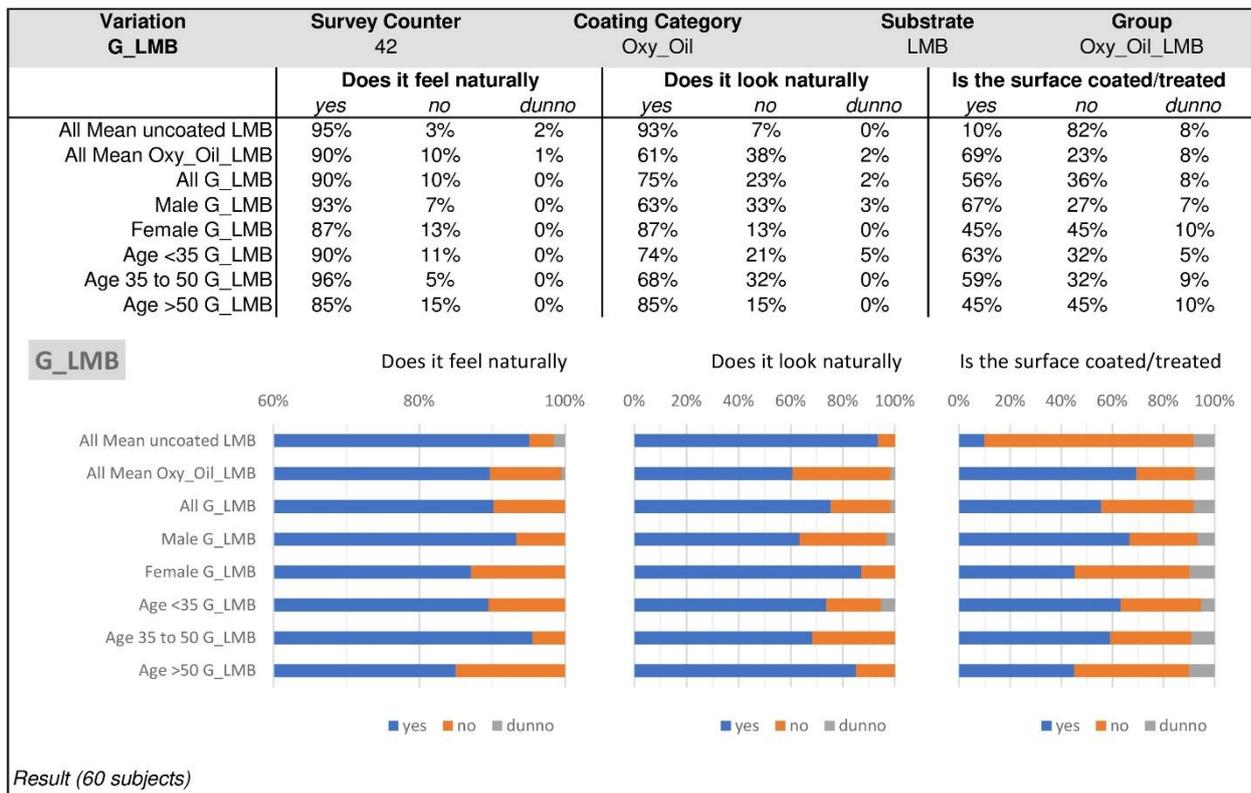


Figure 4: SEM micrographs of brushed oak wood surface with uniform coating films which do not obscure the surface structure

A result from the test person study is shown in Fig. 5. 90 % of the test persons answered that this surface had a natural feel, 75 % replied it had a natural look. Table 1 summarises the results from the test person study and chemical resistance testing leading to an overall assessment which combines these aspects. The majority of the samples that were visually perceived as natural were also classified as haptically natural. There were differences between the types of wood larch, oak and maple but also between the pre-treatment levels sanded, medium brushed and dense brushed. The results were strongly influenced and varied by the individual coating systems where gloss, roughness and film thickness had major influence. Trends were recognized that the brushed larch was felt to be more natural than the sanded larch.



**Figure 5: Result of test person study for coating G on larch medium brushed (G\_LMB)**

**Table 1: Summary of results from haptic survey, chemical resistance testing and the overall assessment**

Benchmark ≥ 65%			Mean of haptic and visual impression				Chemical Resistance F: 1-B, Non-F: ≥ A						Overall Assessment							
Ff.	Coat_ID	Coat_Cat	MS	LS	LMB	OS	OMB	ODB	MS	LS	LMB	OS	OMB	ODB	MS	LS	LMB	OS	OMB	ODB
Non-F	C	Oxy_Oil		63%	77%	57%	69%			B	B	B	B							
Non-F	F			66%	79%					B	B									
Non-F	G			89%	83%					A	A									
Non-F	K					72%	66%	69%				B	A	A						
Non-F	O			59%	64%	73%	66%	54%		A	A	A	B	A						
F	P					52%	35%	34%					1-C	1-C	1-C					
F	Q					42%	52%	47%					1-C	1-C	1-C					
F	R					n.e.	n.e.	n.e.					-	-	-					
Non-F	T					80%	74%	66%					B	B	-					
Non-F	V					84%	60%	57%					A	A	A					
Non-F	Y			74%						A										
F	W	UV_lacquer				37%	75%	54%				1-B	1-B	A						
Non-F	D	UV_Oil		76%	77%	72%	66%			A	1-B	A	B							
Non-F	E			81%	72%	48%	49%			A	A	A	A							
Non-F	L					75%	74%	68%					A	A	A					
Non-F	M			66%	90%					A	A									
F	S					52%	41%	68%				1-B	A	A						
Non-F	X					57%	55%	67%					1-C	B	1-C					
Non-F	A	Wax/Oil-wax		52%	56%	57%	47%			B	A	A	C							
Non-F	U				68%	69%	52%					B	B	B						
F	B	wb_lacquer	58%	92%	91%	78%	80%		B	B	B	B	B							
F	H		36%						B											
F	I		33%						B											
F	J		58%						B											
F	N			32%	43%	16%	16%	11%		B	B	B	B	B						

## CONCLUSIONS

A total of 17 coated wood flooring variants were developed that had a good chemical resistance and also a natural visual and haptic appearance (1 on maple, 7 on larch, and 9 on oak; Table 1). Coating parameters such as gloss, roughness and film thickness that influenced the natural appearance were identified. Surfaces with less gloss and higher roughness were perceived as more natural. With the variants and techniques developed in the Surf~Parquet project, it was possible to produce thin continuous films on the surface despite the strong structuring. Hence, the surfaces were durable and looked natural.

## White clouds – Undesired discolouration of oak wood during kiln drying

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**Keywords:** Oak wood, drying, colour change, raman microscopy

### ABSTRACT

The issue of inhomogeneous colour changes of oak wood during drying processes is well known in wood industry and can cause drastic losses in quality. These colour inhomogeneities, namely darkening from the edges, are frequently also called “white clouds”. Although the phenomenon is well known, the reasons and conditions how and when the colour change occurs are not fully understood or controversially discussed. Own investigations show the connection between discoloration and moisture distribution. To further investigate the background of the colour differences, extractions with hexane, acetate and formate were first carried out and analysed by GC-MS. However, no differences in detected substances could be found. Also, ATR/FTIR spectra of light and dark areas showed no differences in the spectrum. By means of Raman measurements on sections of light and dark areas, indications could be detected. The hypothesis is that tannins in the more humid regions hydrolyse to ellagic acid. Ellagic acid was found preferentially in the lighter areas.

### INTRODUCTION

Oak wood is one of the most valuable European hardwoods, which is used for many high-quality applications, especially in the furniture and flooring industry, where particularly high-quality standards are required. Unfortunately, irregular discolouration can occur during the technical drying process, which reduces the quality and leads to financial losses (e.g. Wassipaul and Fellner 1992; Fortuin et al. 1988). Fig. 1 shows an example of irregular brown discolouration from the edge, colloquially called "white clouds".



*Figure 1: Typical “white clouds” (arrows) surrounded by darker colour at the edges of the cross-section*

The problem is well known and numerous studies are concerned, for example, with the optimisation of drying parameters to avoid this undesirable colour inhomogeneity (e.g. Fortuin et al. 1988; Welling and Wöstheinrich 1995, Stenudd 2005), while other studies uncover the involvement of extractives (especially ellagitannins) in colour reactions (e.g. Koch and Skarvelis 2007, Kisseloff 1993, Wassipaul and Fellner 1992, Wassipaul et al. 1987, Wegener and Fengel 1988). Although the phenomenon is well known, the reasons and conditions how and when the colour change occurs are not fully understood and are sometimes controversially discussed. Especially the responsible processes on the microscale are still largely unknown. In the present work, therefore, an attempt has been made to contribute to the subject matter by means of systematic drying experiments and analytical methods at both, the macro and micro level.

## EXPERIMENTAL METHODS

Oak boards (*Quercus robur* L.) of 65 mm thickness and app. 1800 mm length were used with varying width. Different drying methods such as air seasoning (protected from rain and sun), kiln drying and radio-frequency assisted vacuum drying were applied. Final moisture content was 9% for technical drying. Boards were cut in cross- and longitudinal sections for “cloud”- investigation and wood moisture determination (Fig. 2).



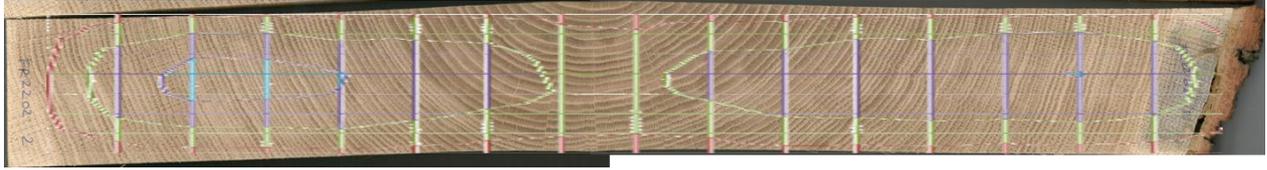
*Figure 2: Sample preparation for “cloud”- investigation and wood moisture determination*

Dark and bright areas were analysed by means of GC-MS (extractions with hexane, acetate and formate) and ATR/FTIR in order to reveal any differences.

For confocal raman microscopy, microsections were prepared with a rotary microtome perpendicular to the main fiber axis. Raman measurements of the microsections were performed with a confocal Raman microscope (Alpha300RA, WITec GmbH, Germany).

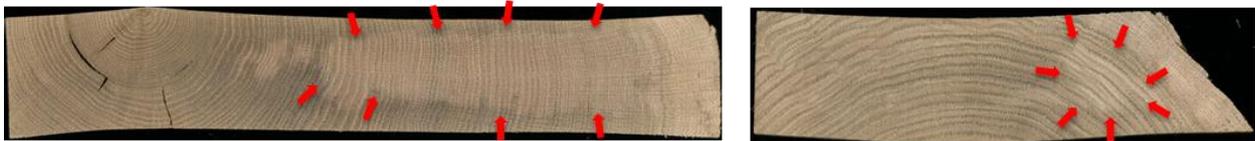
## RESULTS AND DISCUSSION

The air-drying experiments showed a clear correlation between moisture distribution and "cloud shape" (Fig.3). As the drying process progresses, the clouds gradually disappear and a uniform brown coloration is observed across the cross-section.



*Figure 3: Clear correlation between moisture distribution and "cloud shape"*

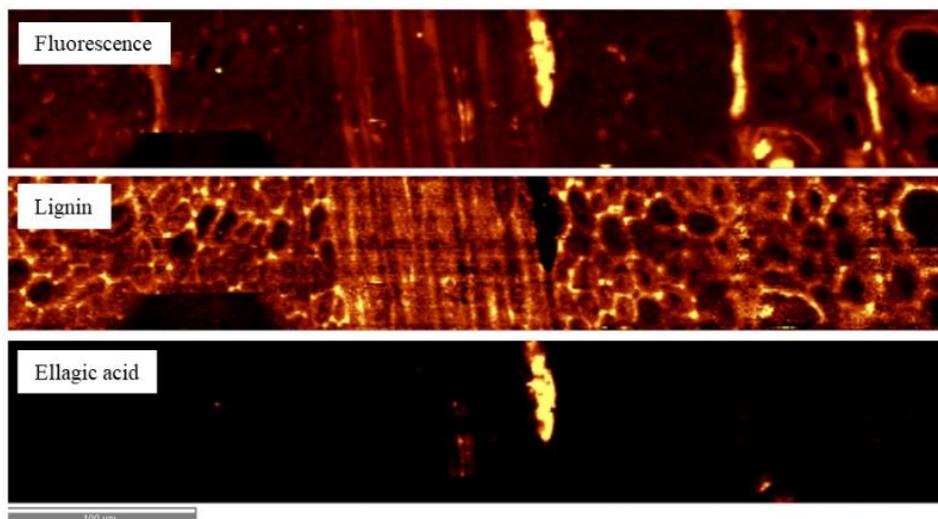
Kiln drying experiments showed that the moisture nests present after previous air drying to approx. 40% wood moisture clearly manifest themselves as "white clouds" after completion of the kiln drying process.



*Figure 4: Sharp "cloud formation" after kiln drying in sequence of previous air drying*

High-frequency vacuum drying resulted in the same results after previous air drying. When drying fresh material (initial moisture 80-90%), however, discoloration could be prevented. However, with a drying time of only 14 days we are also at the edge of possible drying rate even with this technique and first cracks occurred.

While the colour differences between light and dark areas are clearly visible to the naked eye, it is surprisingly difficult to determine the chemical background. To investigate the background of the colour differences, extractions with hexane, acetate and formate were first carried out and analysed by GC-MS. However, no differences in detected substances could be found. Also ATR/FTIR spectra of light and dark areas showed no differences in the spectrum.



*Figure 5: Raman-microscopy pictures of a wood ray at the border of a white cloud with 532nm laser. From top to bottom: fluorescence of the specimen, distribution of lignin and of ellagic acid*

By means of Raman measurements on sections of light and dark areas, indications could be detected (Fig. 5). The hypothesis is that tannins in the more humid regions hydrolyse to ellagic acids. Ellagic acids was found preferentially in the lighter areas. They form during the hydrolysis of ellagitannins under certain temperature and humidity conditions and are deposited in rays, lumens, pits and cell corners. These areas are the main ways of water in the wood and their obstruction restricts water transport on the microscale. However, an unobstructed path is crucial for the homogeneous transport of hydrolysis products during drying. Consequently, the hydrolysis products precipitate as crystalline ellagic acid and are thus removed from the oxidation reaction, leaving these areas lighter and appearing as so-called "white clouds".

## CONCLUSIONS

The formation of unwanted discoloration during the drying of oak lumber, the so-called "white clouds", was systematically investigated. On the macro level, the formation of discoloration could be related to moisture nests present after outdoor pre-drying. On the micro-level it can be assumed that discoloration in oak heartwood is caused by trapped tannin hydrolysis. This results in crystalline ellagic acid and, as a consequence, a significantly reduced brown colouring. The drying rate plays an important role here - but it can only be adjusted within certain technical limits, depending on the drying technology.

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## Effects of cement on lignocellulosic fibres

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**Keywords:** Lignocellulosic fiber, cement, compatibility, hydration, curation.

### ABSTRACT

Lignocellulosic fibers are available throughout the world from different agro-forestry companies which could be utilized for the reinforcement in cement/fiber-based composites. The composites made from the lignocellulosic fibers compositing with the cement could be enhanced with numerous functionality and properties for using in construction and building sector. However, the proper selection of fibre is necessary to ensure better compatibility between the lignocellulosic fibres and cement. This research work investigates about the testing of sugar and tannin content and hydration of the cements with fibres. The Scanning electron microscope (SEM) images was investigated for the checking of fibre and cements existence in the matrix. The SEM analysis has further shown the regular distribution of fibers throughout the composite panels. This research would also help to identify the effects of different additives for improving the compatibility of cements and fibers on the cement bonded fibre boards.

### INTRODUCTION

Recently, considerable efforts are seen with the researchers to develop cement bonded lignocellulosic fibre composites for affordable and fashionable infrastructures. But, the usage of natural fibre-based cementitious materials are still limited for durability issues along with the development of poor resistance against crack formations. So, different investigations are going on develop toughness and ductility of fibre reinforced cement-based composites (Yao et al. 2003, Savastano Jr et al. 2009). However, the incorporation of vegetable fibers (Hasan et al. 2020) could enhance the microstructure interactions between the cement and fibres in matrix. Nevertheless, cementitious composites exist outstanding potentiality for low-cost, superior compressive strength, low maintenance requirements, and attractive appearances (Wei et al. 2016). Furthermore, to fulfill the ever-increasing demands towards the sustainable structural composites, natural fibres (Hasan et al. 2020, Hasan et al. 2020, Hasan et al. 2020) are attracting the interests of the scientists continuously.

Compatibility is another critical problem for the production of fibre-based cementitious composites. The inorganic alkaline chemical component (cement) contains hydroxyl groups in the surface. On the other hand, fibres originated from the hardwood exhibits strong inhibition against the cement. Such problems are generated both in the cement setting and curation stages. However, inhibitory problems are quite less during the curation period of the hardwood (Pehanich et al. 2004). Exothermic hydration processes are strongly affected by the extractives of fibres with ordinary Portland cement which is the main reason for the incompatibility problems of wood fibres and cements (Castro et al. 2018). Although plant fibres are comprised of many chemical compounds but the sugar and tannin contents are mainly responsible for the inhibition of wood fibre and cement hydration and thus consequences on the strength. So, we have conducted sugar and tannin content tests of our lignocellulosic fibres extracted from different species of Hungarian plants and found our results were quiet satisfactory that no need any additional pretreatments to remove the impurities.

The effects on different ratios of cement/fibre mixing, storage environment for hydration temperature, time for hydration has significant influences for the reduction of hydration temperature and time. The usage of plant-based natural fibres has been enhanced for minimizing the costs of construction materials in terms of

economic aspect to meet the consumer demands. However, the constituents of wood fibre and cement has made it challenging for the hydration problems and presence of impurities (sugar and tannin content). So, we have conducted this research to optimize these problems through analyzing the hydration of cements with different chemical ingredients. The SEM images were also analyzed to find out the fibre cement appearances in the matrix before and after the applying the loads.

## MATERIALS AND METHODS

### *Materials*

The lignocellulosic fibres used for this study was kindly supplied by a local Hungarian company (Kronospan-MOFA Hungary Ltd.). The fibers were used without any further pretreatment. The cement was kindly supplied by another local company of Sopron, Hungary (OPC CEM I 42.5). The chemical ingredients (water glass, montmorillonite, and CaCl<sub>2</sub>) were purchased from Sigma Aldrich.

### *Methods*

The sugar and tannin content of the fibres were investigated before starting the fibre board productions. The hydration rate of the chemical, fibre, and cement was also checked to ensure about the suitability of the fibre composites manufacturing. The cement bonded fibre boards were prepared by using compression moulding machine (G. Siempelkamp GmbH, Germany). Two fibre boards (WCF (water glass, cement, and fibre) 1 and WCF 2) were prepared from water glass (0.05 and 0.06), ordinary Portland cement, fibre (1), and water by varying the cements (2.5 and 3.5) in the matrix proportion. The dimensions of the composite boards were 400 mm X 400 mm X 12 mm. The applied pressure was 18.5 MPa for 24 h at normal temperature. The composited panels were then cured for another 28 days in room temperature. The SEM images were also taken by using Hitachi S-3400N instrument (Japan) at accelerating voltage of 20.0 kV for fibre boards and 20.0 kV for fractured composites.

## RESULTS AND DISCUSSION

The sugar and tannin contents of the fibres were checked before starting the composites productions. The assessed tannin content of the fiber was around 0.25% which is within the standard range (upto 0.4% is considered as accurate). On the other hand, sugar content of the fibres were less than 0.5% according to our assessment which ensures that the fibres did not contain any unacceptable impurities that could appeal for further pretreatment (Sudin and Swamy 2006, Tibor L. Alpar- Éva Selmeczi 2012). It may be that the fibre supplying company has applied a boiling treatment before the production of fibers.

**Table 2: Temperature versus time characteristics of different cementitious materials**

Samples	T <sub>(max)</sub> (°C)	T <sub>(min)</sub> (°C)	t <sub>(max)</sub> (min)	t <sub>(min)</sub> (min)	$\frac{dT(T_{max}-T_{min})}{dt(t_{max} - t_{min})}$
<b>MPCF</b>	26.1	20.3	9	1440	0.0041
<b>WCF</b>	23.6	20.2	5	1440	0.00237
<b>CPCF</b>	21.7	20.2	7	570	0.00266

When the water was added to the different samples (MPCF, WCF, and CPCF) the chemical reaction is started which brings a structural changes in the cementitious paste which turns to hard and rigid from the fluid mass with a generation of significant amount of heat (Wei et al. 2000). The temperature changing rate (dT/dt) and temperature versus time study were investigated for MPCF (montmorillonite + PDDA + cement + fibre), WCF, and CPCF (CaCl<sub>2</sub> + water glass + cement + fibre) as shown in Figure 6 and Figure 7. The Figure 6 shows the characteristics of temperature and corresponding time. The maximum temperature (26.1 °C) was found for MPCF and the time required to reach this temperature was 9 min. However, the highest temperature for WCF was 23.6 °C achieved at 5 min, and 21.7 °C for CPCF achieved at 7 min. It seems that, the hydration of cement requires the minimum time for WCF (WCF<CPCF<MPCF), whereas the highest time needed for MPCF. The addition of montmorillonite and CaCl<sub>2</sub> may increase the hydration time a little bit compared to the water glass. On the other hand, dT/dt value of WCF

(0.00237) is also comparatively lower than both CPCF (0.00266) and MPCF (0.0041). Some of the similar phenomenon was also reported by other researcher (Jo and Chakraborty 2015).

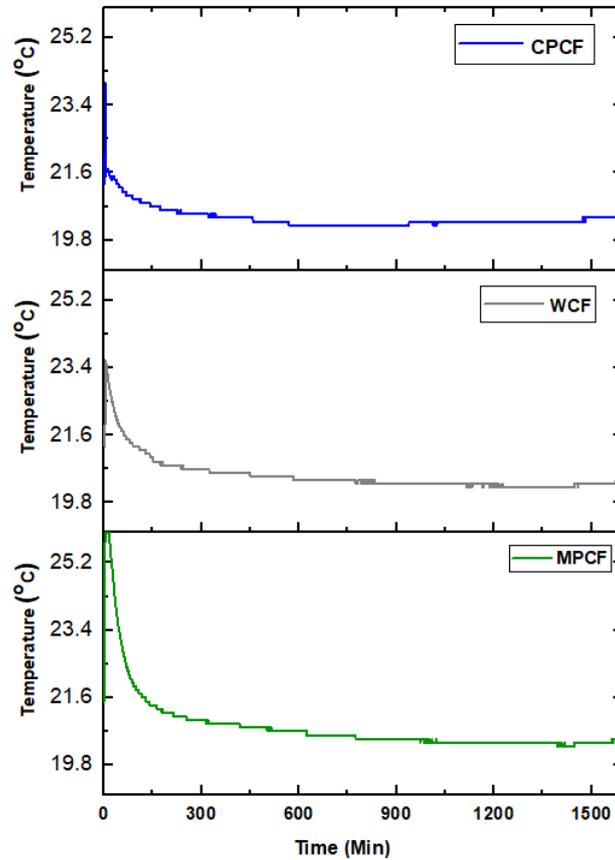


Figure 6: Time versus temperature curves for assessing the effects of different additives on cement hydrations

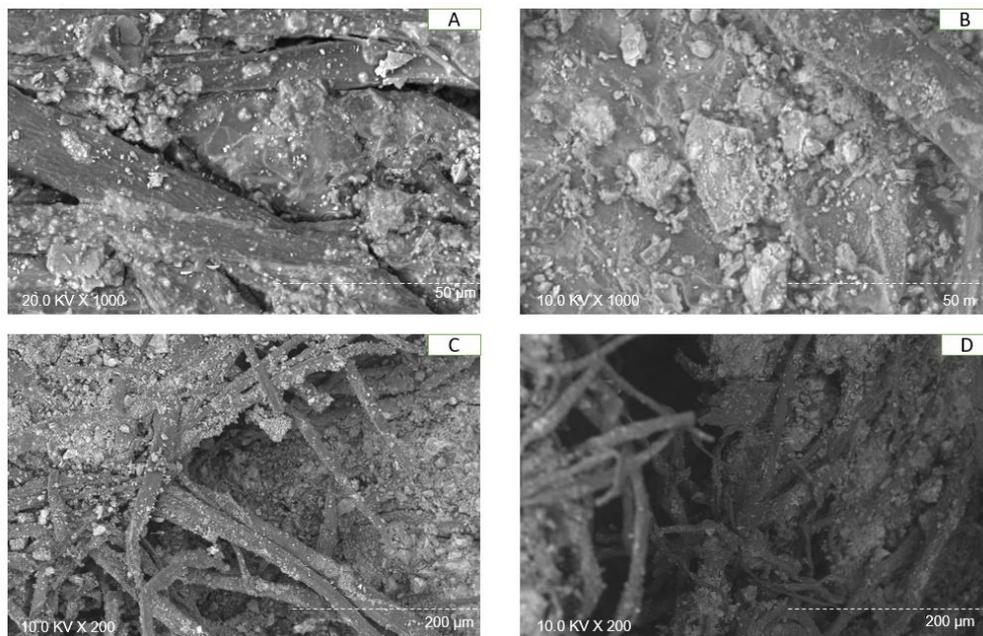


Figure 7: SEM images of (A) fibre board 1 (WCF), (B) fibre board 2 (WCF), (C) fracture of fibre board 1(WCF), (D) fracture of fibre cement board 2(WCF)

The SEM images (Figure 7) were used to visualize the samples for observing the structure and defects of the produced composite panels. The Figure 7 (A and B) exhibits the presence of uniformly distributed lignocellulosic fibres in the cementitious matrix. The Figure 7 (A) shows more presence of fibres in the composites maybe for the presence of less cement (2.3) than the Figure 7 (B) entailing 3.3 gm of cement. However, Figure 7 (C and D) shows the pull out and breakage of the matrix in composite system when the stress was applied. The similar phenomenon were also described by other researchers for wollastonite fibre-based cement composites (Tichi et al. 2019).

## CONCLUSIONS

The sustainable green fibre cement composite has extremely high potentially to be applied for different structural applications. An investigation regarding the fibre cement composites compatibility is reported in this research. The sugar and tannin content of the fibre was investigated for finding out the pretreatment necessity. It is found that both the sugar and tannin contents were within satisfactory range that no need for fiber pretreatment. The optimum hydration rate was found through this study. It is seen that, WCF requires the minimum time to reach highest temperature compared to MPCF and CPCF. The SEM images has clearly shown the uniform distribution of fibres throughout the matrix. Afterall, this research would support for future works on developing more innovative, lighter weight, low-cost, and environment-friendly composite panels in building and construction sectors.

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## Effect of harvester logging on timber produced in deciduous stands

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**Keywords:** harvester, logging, performance, selective cutting, working day structure, assortment.

### ABSTRACT

As a result of new developments in technology, harvesters can be used not only in pine forest but also in hardwood stands. This technology is now not only used in clear cutting, increment and selection thinning in Hungary, but also in selective cutting. These harvesters are not only used in state and private forests, but also in protected forests of some national parks. We made measurements in the Órség National Park where selective cutting was done with a high mechanized logging work system. In order to analyze and evaluate the structure of the work day and performance of the harvesters, field surveys were done with continuous time measurement. Besides recording the duration of individual actions, among others the total volume of the timber processed in each cycle and the distances of changeovers were also recorded.

### INTRODUCTION

As a result of new developments in technology, harvesters may no longer be confined to softwood forests only. Several studies carried out in black locust, Turkey oak, beech and oak stands have justified the use of these machines in hardwood stands. Evaluating the results of the cost and time analyses we concluded that harvesters are more efficient in several cases compared to traditional wood cutting with chainsaws.

### EXPERIMENTAL METHODS

#### *Analysis and evaluation of the work with harvesters*

Nowadays multi-operational logging machines work in both hardwood and softwood stands. In Hungary, depending on conditions of terrain, some of this equipment is primarily used in hardwood stands, while other types are almost exclusively used in softwood forests. Based on the foreign results in efficiency and productivity of harvesters, there is no doubt that these machines should be the first choice for domestic softwood forests too. However, their use in domestic hardwood stands raises many questions. In the past few years multi-operational logging machines have already been used for logging domestic forests (in black locust, alder, hybrid poplar, Turkey oak, hornbeam-oak, beech, hornbeam-Scots pine, spruce, Scots pine and black pine stands). Clearcutting, thinning, preparatory cutting and sanitary cutting, as well as selective cutting were all done with these machines.

In order to analyze and evaluate the structure of the work day and performance of the harvesters, field surveys were done with continuous time measurement. Besides recording the duration of individual actions, the total volume of the timber processed in each cycle and the distances of changeovers were also recorded. During the survey, the following types of actions were distinguished (Horváth A., 2012; Horváth A., 2015):

- Felling (F): equals the time required for the machine operator to place the harvester head onto the base of the tree, using the manipulator arm, and felling, preassembly, debranching of the log as well as the conversion into assortments and piling by assortments.
- Changeover (C): is the duration of machine displacement.
- Felling only (FO): time spent on logging very thin or poor quality (e.g. completely rotten) logs, which do not yield valuable assortments.

- Arranging branch material (B): transfer and rearrangement of branches obstructing the path of logging.
- Arranging timber (T): transfer and arrangement of timber stacks obstructing the path of logging.
- Rest period (R): time for meeting personal needs.
- Troubleshooting (TH): time for fixing technical defects in the machinery.
- Waiting (W): other time losses (e.g. phone calls).

### **Selective cutting using the Ponsse Ergo 6WD harvester**

The selective cutting in a 97-year-old, 3,21 hectare total area of hornbeam – beech – Scotch pine mixed stand (Figure 1) was done using a Ponsse Ergo 6WD multi-operational logging machine. The forest area, is protected, located in the Őrség National Park and is also part of the NATURA 2000 network. According to the data of the management plan – depending on the tree species –, the average height of the trees was 17 - 26 m, and average diameter at breast height was 20 - 40 cm (Table 1). Before the logging, workers of the forest management unit had marked the trees intended for cutting with paint. The machine operator converted the logs into assortments as follows: 3,1 m, 4,1 m, 5,1 m, 6,1 m logs and 2,0 m pulpwood for boards from pine, as well as 2,0 m household firewood from beech and hornbeam.



**Figure 1: Hornbeam – beech – Scotch pine mixed stand, Apátistvánfalva 28C**

**Table 1: Data of management plan, Apátistvánfalva 28C**

Number	Canopy	Tree species name	Mixture rate [%]	Mixture mode	Aver. age [year]	Aver. height [m]	Aver. diam. [cm]	Canopy closure [%]	Blasa area [m <sup>2</sup> /ha]	Wood stock [m <sup>3</sup> /ha]	Wood stock in subcompartment [m <sup>3</sup> ]
1	Upper	Beech	23	Main species	77	20	25	77	8	78,01	250
2	Upper	Beech	4	Spread	97	26	40	77	1	20,00	64
3	Upper	Scotch pine	46	Spread	97	22	33	77	15	166,01	533
4	Upper	Pedunculate oak	9	Spread	97	25	39	77	3	32,99	106
5	Upper	Hornbeam	18	Spread	77	17	20	77	4	36,01	116
6	Lower	Hornbeam	100	Main species	37	15	17	25	6	45,02	145
<b>Total</b>									<b>37</b>	<b>378,04</b>	<b>1 214</b>

## RESULTS AND DISCUSSION

### Working day structure

The field survey and data collection were done in 3 days (708,17 minutes). During the period of the measurement (Table 2, Figure 2) 54,7% of the operating time was spent on felling trees, 21,3% was for changeovers, and around 4,2% of the time was spent arranging the branch material. This type of harvesting involves a relatively high frequency of changeovers. The average changeover distance was 6,8 m, and the average time needed for changeovers 0,7 min. The proportion of rest period was 15,5%.

Table 2: Data of action items, Ponsse Ergo 6WD harvester

ACTION ITEMS	Σ TIME [MIN]	RATE [%]	ELEMENT [PIECE]	AVER. TIME
FELLING (F)	387.65	54.7	302	1.28
CHANGEOVER (C)	151.16	21.3	216	0.70
FELLING ONLY (FO)	21.00	3.0	31	0.68
ARRANGING BRANCH MATERIAL (B)	29.59	4.2	37	0.80
ARRANGING TIMBER (T)	4.25	0.6	5	0.85
REST PERIOD (R)	109.77	15.5	8	13.72
TROUBLESHOOTING (TH)	4.33	0.6	1	4.33
WAITING (W)	0.42	0.1	1	0.42
<b>TOTAL (Σ)</b>	<b>708.17</b>	<b>100.0</b>		

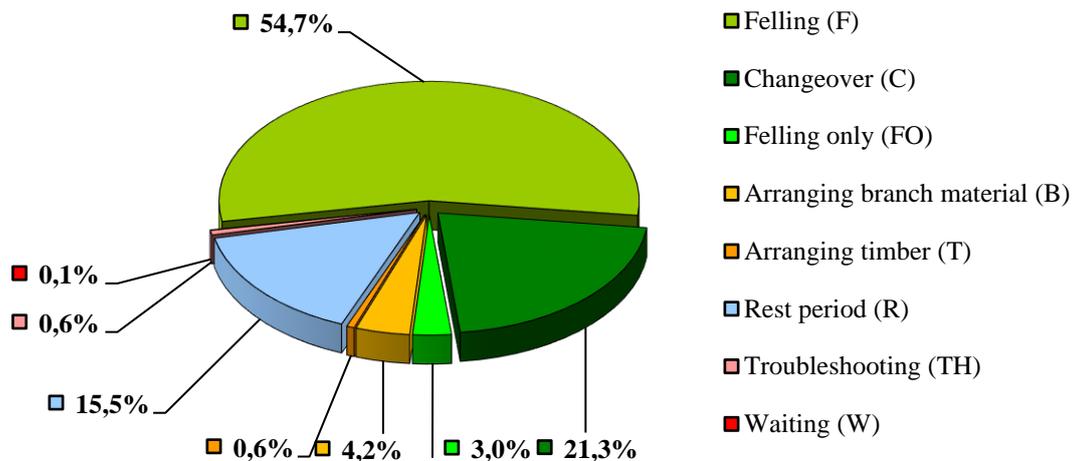


Figure 2: Working day structure of Ponsse Ergo 6WD harvester (Rátky, 2019)

### Performance of harvester

The volumes of harvested and processed timber (146,6 m<sup>3</sup>) and the duration of the single actions were considered to estimate performance values. The hourly performance of the Ponsse Ergo 6WD multi-operational logging machine (working time) was 12,4 m<sup>3</sup>/h. The shift performance in productive time was 118,5 m<sup>3</sup>/shift (Table 3).

Table 3: Performance of the Ponsse Ergo 6WD harvester (Rátky, 2019)

Performance		[m <sup>3</sup> /h]	[m <sup>3</sup> /shift]
Logging time	(F+C)	16,3	130,6
Productive time	(F+FO+C+B+T)	14,8	118,5
Working time	(Σ)	<b>12,4</b>	<b>99,4</b>

Altogether 331 trees were cut, which were 46,2% of Scotch pine, 39.6% of hornbeam, 12.4% of beech, 1.2% of aspen and 0.6% of pedunculate oak. The cut 146.6 m<sup>3</sup> of wood was made up of 1813 assortments (Figure 4).

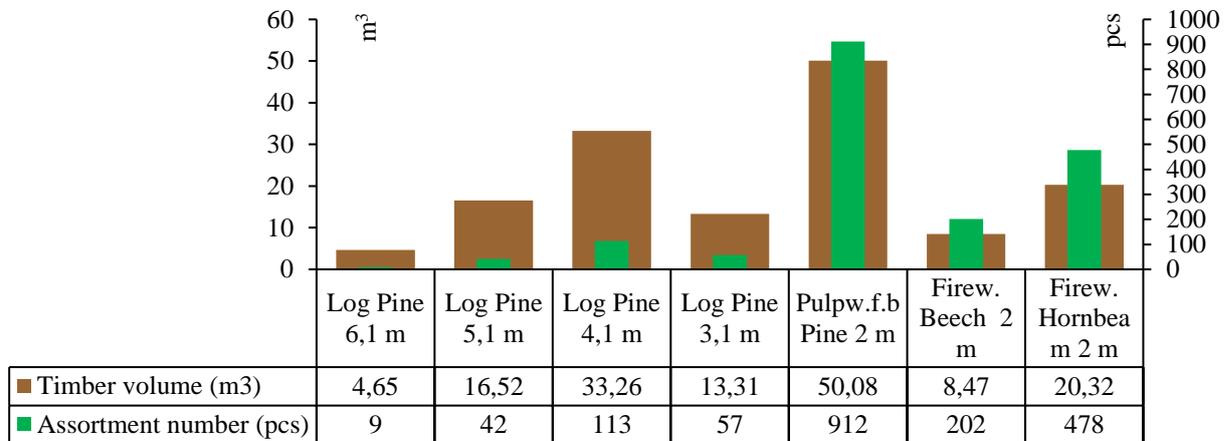


Figure 4: Assortment structure

## CONCLUSIONS

Regarding optimal technological developments, logging done with harvesters can also be introduced to selective cutting and not just clearcutting and thinning. This however requires higher level professional attention and control.

## ACKNOWLEDGEMENTS

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## Thermally activated building system – innovative application for beech

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**Keywords:** *Fagus Sylvatica*, Norway spruce, thermal component activation, building mass, building component simulation

### ABSTRACT

A practicable and cost-effective way to store energy and thus facilitating the use of renewable energy is the utilization of building mass as thermal storage. This principle is used and approved for concrete, the current study deals with wood as alternative storage mass and the investigation on the utilization of a thermally activated building system (tabs) using hard wood elements.

Different wood-based materials produced from soft wood as well as from hard wood had been selected for this study. Using the thermal conductivity values of these materials derived from literature and from previous investigations, some parameter studies applying steady-state simulations had been performed to develop different prototypes of slab elements. A two-dimensional steady-state building component simulation was carried out with HTflux, a simulation tool for thermal analyses. The steady-state simulations allow to draw conclusions on the attainable heat flux densities and surface temperature fluctuation as a function of the mounting depth of the piping (energy source).

The steady-state simulation results show that wood can be used for thermally activated wall or ceiling elements despite its low thermal conductivity. An innovative combination of soft and hard wood layers results in a higher heat flux density compared to merely softwood construction elements. These results show a high potential of hard wood as wood-based material improving the functionality of thermally activated building systems.

First results show that beech or other hard wood species could be useful for the development of tabs in timber constructions. These results may foster the development of innovative hard wood materials for such functional applications.

### INTRODUCTION

In recent years solid timber constructions became constantly more importance in construction, expansion and renovation of buildings. This might be beside ecological considerations a result of a possible short assembly time of industrial prefabricated wooden elements due to the high level and quality of the prefabrication process. The use of wood-based materials is still increasing for the timber construction sector (Paulitsch and Barbu 2015). This study deals with the investigation of the integration of a room climate conditioning function of wood-based materials by using tabs.

However, the good insulation properties of wood could interfere this approach and this needs further considerations. The thermal conductivity values differ from timber construction materials as well as between various wood species (Niemz 2007, Niemz and Sonderegger 2011) and within one wood species between the fibre directions – radial/tangential versus longitudinal. In radial/tangential fibre direction Norway spruce (*Picea abies*) has a thermal conductivity value around 0.100 (W/mK) and beech (*Fagus sylvatica*) around 0.176 (W/mK) (Niemz 2007). In comparison reinforced concrete has a 20 times higher thermal conductivity than spruce (2.300 W/mK) (www.beton.wiki/).

The comparison of the specific heat capacity shows higher values for wood – 2100 versus 880 (J/kgK) but for the use of the materials as a tabs concrete yields an approximately 2 times higher heat storage value due to the higher density – 475 (660) versus 2300 kg/m<sup>3</sup> (www.beton.wiki).

The introduction of cross-laminated timber (CLT) has become a successful story of an innovative product development. Smart functionalisation as heating and cooling may be a further step in the development of wooden elements made of CLT. Brigola (2010) and Mindrup (2019) analysed the different concepts of wood-based panels and CLT products for the potential of thermally activated timber elements by using a system of different milled air holes in the material, where the air is flowing through. They concluded the usability of thermally activated solid timber elements as surface heating is possible. However, the activated layer through air flow is nearly needed on the surface (e.g. 0.015 m). Wehsener et al. (2016) analysed different properties of multi-layer solid wood panels with a thickness between 0.22 and 0.26 m and with integrated pipes for the heating and cooling. Their results of the combination of wood with a high density (e.g. hardwood) and the pipes with a small overlapping of 0.004 m was shown an optimum. However, with this low mounting depth of the air holes and/or even pipes, the possible storage of energy in the building mass as thermal storage and the usability of this energy is difficult. On the other hand, a practicable and cost-effective way to store energy and thus facilitating the use of renewable energy plays an important role in the future. This principle is used and approved for concrete, the current study deals with wood as alternative material.

## EXPERIMENTAL METHODS

### *Material and structure of the constructional elements*

For the investigation industrial fabricated cross-laminated timber (CLT) from Norway spruce was used as basic material. For the modification of the prototypes, beech wood was applied at different positions of the CLT element (Table 1).

*Table 1: Description of the different structure of constructional elements*

Variant	Thickness of CLT [m]	Ratio of Spruce [%]	Ratio of Beech [%]
1	0.16	100.0	0.0
2	0.16	100.0	0.0
3	0.16	81.3	18.7 <sup>a</sup>
4	0.16	62.5	37.5 <sup>a</sup>
5	0.16	81.3	18.7 <sup>b</sup>
6	0.16	62.5	37.5 <sup>b</sup>

<sup>a</sup>beech sample with growth ring position (radial-tangential) <sup>b</sup>beech sample with growth ring position (axial)

For the prototype production the CLT elements were cut apart by band saw so that the piping (energy source) could be installed at the appropriate depth. The pipes were cut into the CLT element using a semi-circular milling cutter. Subsequently, the pipes and the wooden parts (spruce and/or beech) were pressed onto the CLT element using PU adhesive (Fibcon 15, beko) so that the overall thickness of the element of 16 cm was achieved. For curing and acclimatise, the prototypes were stored at 23 °C and 50 % relative humidity.

### *Thermal conductivity*

The thermal conductivity of the different beech samples was measured according to the ÖNORM EN 12667 (2001) with the  $\lambda$ -Meter EP500e from the Lambda-Messtechnik in a conditioned room at 23 °C and 50 % relative humidity. The  $\lambda$ -value was determined at 10 °C, 25 °C and 40°C and provided the basis for the numerical simulation.

### *Simulation*

The numerical simulation was performed with HTFlux software for different prototypes of the thermally activated elements built up with parts of Norway spruce and beech. For the parameters of the simulations standard reference values from the HTFlux software were selected and are shown in Table 2. Only the

thermal conductivity values of the beech samples were measured with the  $\lambda$ -Meter EP500e for the different growth ring directions.

**Table 2: Parameters for the numerical simulation processes**

Variant	Thermal conductivity	Heat capacity [J/(kgK)]	Density [kg/m <sup>3</sup> ]
Screed	1.33	1080	2000
Impact sound insulation	0.035	1030	68
Fill	0.70	100	1500
Norway Spruce	0.13	1600	500
Beech <sup>a</sup>	0.1763	1600	724
Beech <sup>b</sup>	0.2874	1600	700
PE-X	0.41	2300	950

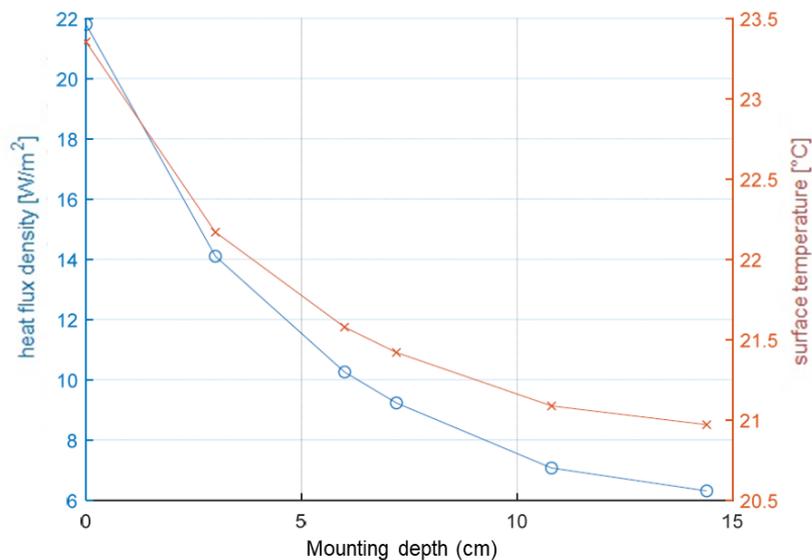
<sup>a</sup>beech sample with growth ring position (radial-tangential) <sup>b</sup>beech sample with growth ring position (axial)

For the present study all parameters stated are referred to 12 % MC as well as we neglected the influence of temperature as the considered temperature range is very small. The influence of MC and density is levelled for the simulations but will be investigated for the laboratory tests of the prototypes.

## RESULTS AND DISCUSSION

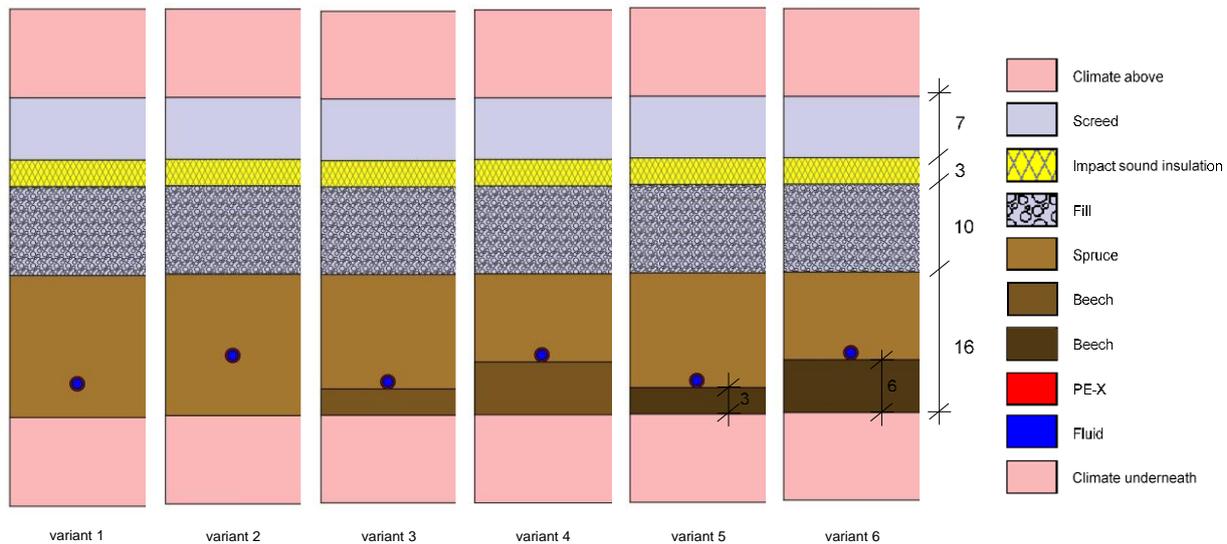
Fig. 3 shows the results of the heat flux density and the surface temperature of the thermally activated CLT element for various mounting depths of the piping (energy source). The heat flux density describes the heat transferred per surface area and time. The higher the values the more efficient is the thermally activated system in terms of thermal output. As surface temperature is limited due to comfort aspects – should not exceed room temperature more than 4 °K –and therefore flow temperature and as a function of it thermal output is limited. The general thermal efficiency of the tabs in situ has to be defined incorporating the heat demand due to the thermal quality of the building.

The best results for tabs made of wood (CLT) regarding heat flux density and applying a flow temperature of 30 °C were calculated for mounting depths of 0.003 and 0.006 m behind the inner surface compared to higher depths (see figure 1). Regarding surface temperature there is still some buffer to higher temperatures in order to increase heat flux density without exceeding surface temperature. For further analysis and laboratory tests only, this mounting depth were used to build the different prototypes.



**Figure 1: Comparison of heat flux density and surface temperature between various tabs overlapping**

The overall structure of the components for the prototype slabs is shown in Fig. 2. These systems were used for the further simulation processes and for laboratory tests. Different prototypes with various depths of the piping and different wood construction elements as well as different wood species (Norway spruce and beech) and fibre directions were developed. On the one hand, the material behaviour was simulated to gain an insight of their potential for thermal activation of building elements. On the other hand, based on the simulation results, selected prototypes will be built up for the validation concerning heat flux density and surface temperature. Also, wood related challenges as inhomogeneous moisture content distribution as well as swelling and shrinkage will be analysed in a further study.



**Figure 2: Overview of the structure of the different construction element**

Fig. 3 shows the results of the simulations on the inner surface of the slab or building element. Differences in surface temperature and heat flux density can be perceived in the different structures of the variants. Flow temperature was 30 °C for all simulations.

The basic variant 1, consisting only of a CLT element with 0.03 m mounting depth of the piping, shows a heat flux density of 14 and a surface temperature of around 22 °C. The simulation results of variant 2, consisting only of a CLT element with 0.06 m mounting depth of the piping, depict lower values of both parameters. It can be assumed that the 0.06 m mounting depth of the tabs is the limit for the utilization of thermally activated wooden building systems (c.f. Fig. 1) at this flow temperature.

In variants 3 and 4 beech wood layer with a fibre orientation (radial-tangential) parallel to the fibre orientation of the slab were applied for the functionalisation of the CLT element. The heat flux density and surface temperature of both variants were higher compared to the elements without beech (variant 1 and 2) as seen in Fig. 3. The beech wood influences positively the results of both mounting depths (0.003 and 0.006 m) due to its higher thermal conductivity and density.

Exchanging the beech wood layer with parallel fibre orientation with beech wood layer with cross-grain fibre orientation (fibre orientation orthogonal to the fibre orientation of the slab) the heat flux density and surface temperature increase further compared to all other variants.

For an optimized and preferable high thermal output, it could make sense to introduce beech wood layers in order to gain a higher thermal output at low flow temperatures and to keep the stress for wood especially regarding swelling and shrinking as low as possible.

Also, a new interpretation of wood-based materials (e.g. cross-grain surface) can boost a development of beech wood products for functionalisation of thermally activated building systems.

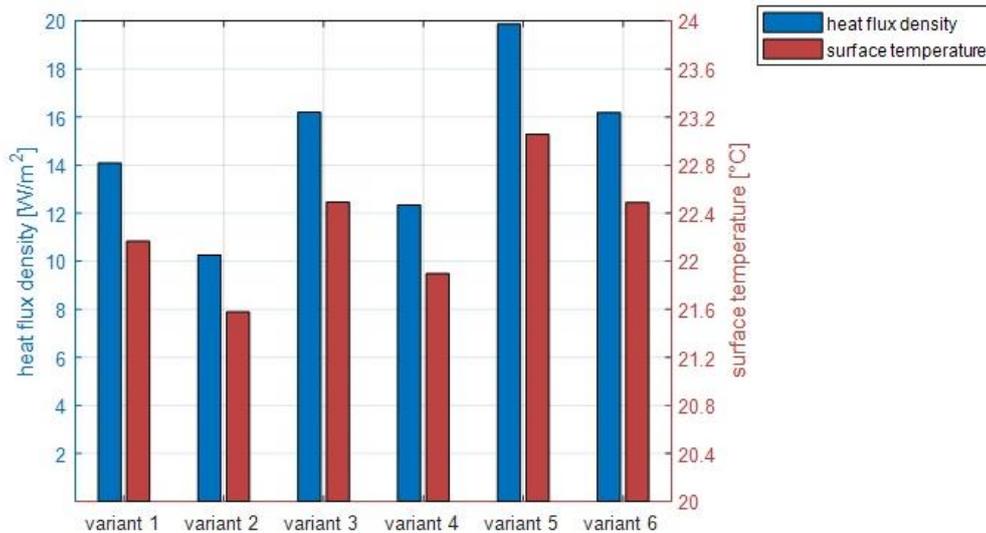


Figure 3: Results of the HTFlux simulations for the different variants

## CONCLUSIONS

The steady-state simulation results show that wood can be used for thermally activated wall elements despite its low thermal conductivity. Through optimising the mounting depths of the piping enhanced results of heat flux density and surface temperature were achieved. Also, the use of beech wood as an additional layer (e.g. 0.03 or 0.06 m thickness) has positive effects on the thermal potential of the developed slab prototypes. Based on these findings, the prototypes will be built up for the validation of the steady-state simulations results. In the ongoing study wood related challenges as, inhomogeneous moisture content distribution as well as swelling and shrinkage will also be investigated. However, the results of the simulations already show the potential of hardwood (e.g. beech) for the functionalisation of thermally activated building systems. This application could be a future area of use for hardwoods. Nevertheless, additional studies are required to transform the results from laboratory to industry.

## ACKNOWLEDGMENT

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## Durability, chemical composition and moisture performance of pubescent oak (*Quercus pubescens*)

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**Keywords:** anatomy, durability, extractives, moisture performance, resistance model, water uptake, white rot, heartwood, sapwood

### ABSTRACT

Forests are under great pressure due to climate changes. Among various types of oak species, pubescent oak (*Quercus pubescens*) will likely gain ecological and economic importance in the region south of Alps. Although this wood species is well known, there are not much data available about chemical properties and durability. A detailed analysis of extractives and durability against wood decay fungi was performed. The results of the chemical analysis revealed that heartwood contains up to 20 % of the extractives, with considerable amounts of phenols, including a fairly high concentration of gallic acid. Anatomical structure, with high frequency of tylosis, resulted in good water exclusion efficacy. The presence of biologically active extractives and superior water exclusion efficacy resulted in good durability, as shown by basidiomycetes tests.

### INTRODUCTION

The share of tree species in natural forests is, in general, a function of climatic (temperature, precipitation amount, precipitation distribution), pedological and topographic parameters, among others (Hanewinkel et al. 2013). Forests are under intense pressure due to global climate changes and from the ensuing increases in abiotic and biotic hazards (Kurz et al. 2008). This pressure will result in biome shifts, which will result in substantial ecological issues and will have considerable economic consequences, including income losses to forest owners and changes in raw material supply for the wood processing industry (Hanewinkel et al. 2013). The results of the model (Hanewinkel et al. 2013) show that, by 2100, between 21 % and 60 % (mean 34 %) of European forests will be suitable only for a Mediterranean oak forest type. This type of forest is thus considered to be a winner of global warming (Hanewinkel et al. 2013). In the Mediterranean area, the genus *Quercus* includes 25-30 species, belonging to various subgenera and sections (Denk et al. 2017). Remarkable morphological and ecological differences characterize this group. *Quercus pubescens* belongs to the white oak group (subgenus *Quercus*, section *Quercus*). Pubescent or downy oak (*Quercus pubescens* Willd.) is particularly important, and has a broad distribution range, from western to eastern Europe (Todaro et al. 2015). *Q. pubescens* is a deciduous or semi-deciduous tree, with the ability to withstand moderate summer drought (Mrak et al. 2019). *Q. pubescens* has developed various mechanisms and adaptations to survive in such a drought-prone environment (Lavrič et al. 2017). Since the amount of pubescent oak is likely to increase in the future (Hanewinkel et al. 2013), it is of great commercial interest to determine the relevant properties of this wood species, which will enable the development of new uses of wood. Currently, the wood of pubescent oak is hardly considered to be industrial wood and is mainly used for energy production. In the past, it was utilized for railway sleepers,

while nowadays it is occasionally used for low-added-value products such as carpentry, or packaging (Todaro et al. 2015). To develop uses of pubescent oak in outdoor applications, the durability of pubescent oak wood has to be determined. Standard EN 350 (CEN 2016) defines the durability of heartwood of several oak species: *Q. alba* (durability class: 2-3), *Q. cerris* (durability class: 3), *Q. robur* (2-4) and *Q. rubra* (3-4). As can be seen from the presented data and cited literature, oak durability is highly variable (Humar et al. 2008; Meyer et al. 2014). One of the indicators that affects the durability of oakwood is density and associated ring width (Humar et al. 2008). Oakwood with narrow rings has durability similar to beechwood (Humar et al. 2008), that is classified as non-durable wood according to EN 350 (CEN 2016). However, better durability has been determined for oakwood with wider rings. Since there are not many durable wood species in Europe, a wood species with high durability has good market potential. Because no durability data on pubescent oak have been reported, not in the standards and nor in the scientific literature, respective issue was addressed in the present study.

## MATERIAL AND METHODS

The research was performed on pubescent oak (*Quercus pubescens*) wood. The wood originated from the sub-Mediterranean phytogeographic region, Podgorski Karst plateau (45°32'56.3"N, 13°54'36.1"E, 430 MAMSL), a karst region in south-western Slovenia. The sub-Mediterranean climate at the site is characterized by cold winters (T<sub>Jan</sub> = 2.9 °C, the period from 1992 to 2017) and dry, hot summers (T<sub>Jul</sub> = 21.5 °C, the period from 1992 to 2017). Its uneven distribution diminishes the effect of high annual precipitation (1300 mm per year, the period from 1992 to 2017) throughout the year. The diameter at breast height of the sampled trees was between 20 and 25 cm; age was about 60 years and height about 10 m. Rotation cycles for this type of trees in between 40 and 100 years. The three trees were cut in August of 2017. Samples consisted of sapwood, mature (recent) and juvenile (old) heartwood. The wood used for analyses was without visible signs of decay, insect damage or blue staining. For the majority of the analyses, samples of the following dimensions were used 1.5 × 2.5 × 5.0 cm<sup>3</sup>. The average tree-ring width was between 0.5 and 2 mm. For reference, beechwood (*Fagus sylvatica*) samples were used.

Scanning electron microscopy (SEM) was performed to detect detailed anatomical features of the three-dimensional structure of the wood. Sample were dried and coated with gold (Q150R ES Coating System Quorum technologies, Laughton, UK) for 30 s with 20 mA intensity. SEM micrographs were then taken at high vacuum and low voltage (between 5 and 12.5 kV). A large field (LFD) and a concentric back scattered (CBS) detectors were used in an FEI Quanta 250 SEM microscope (Hillsboro, Oregon, United States).

To determine the extractive content, soluble components were extracted from wood using an automated extraction system Büchi B-811 (Büchi Labortechnik AG, Switzerland). The same specimens were used for subsequent density assessment. In the study, samples were extracted with an ethanol:water (ratio 1:3) based mixture. Extractive content was determined on 30 specimens, originating from three trees. Three extractions (12 h each) performed according to the standard Soxhlet procedure. Samples of sapwood (SW), outer/mature heartwood (MHW) and inner/juvenile heartwood (JHW) were taken in a radial series from each of the stem discs sampled. The woodblocks were then cut to smaller pieces and oven-dried at 45 °C for 48 hours. For the purposes of extraction, the wood samples were disintegrated with a Retsch SM 2000 cutting mill using a 1 mm sieve. A day before extraction, ground samples of SW, MHW, and JHW were put on the shelves of a Telstar LyoQuest lyophilizer, where they were freeze-dried at 4 Pa and -82 °C for 24 hours.

Ground and freeze-dried wood of pubescent oak were extracted with a Thermo Scientific system for accelerated solvent extraction (ASE) (Dionex ASE 350 system). The extraction was performed sequentially, firstly with a less polar solvent and afterwards, a more polar solvent, i.e., according to the idea presented in the report of (Willför et al. 2004). In this investigation, cyclohexane was used for extraction of lipophilic compounds, while water and a mixture of acetone and water (70:30, v/v) were selected for extraction of hydrophilic extractives. The contents of lipophilic and hydrophilic extractives were determined gravimetrically. Ten mL of extract was oven-dried at 105 °C to constant mass. Results were expressed as contents in milligrams of extracted matter per gram of freeze-dried wood (mg/g dw). Before UV-Vis and HPLC analysis, the wood extracts of pubescent oak were dried in a vacuum at 0.01 MPa. The dry extract was then dissolved in methanol. Samples were filtered through polyamide filters into vials. Before analysis, all the extracts were stored at -24 °C. Total phenols in extracts were analysed

colorimetrically using a Folin-Ciocalteu phenol reagent, as proposed by Singleton and Rossi (Singleton and Rossi, 1965). The protocol for measuring total phenols was slightly modified and has already been described (Vek et al. 2019). Acetone-water and water extracts of pubescent oak were further analysed on a Thermo Scientific system for high-performance liquid chromatography (Accela HPLC) (Poljanšek et al. 2019; Vek et al. 2019). Three  $\mu\text{L}$  of the sample was injected into a loop of the system. The separation was carried out on a Thermo Scientific Accucore octadecylsilyl (C18) column.

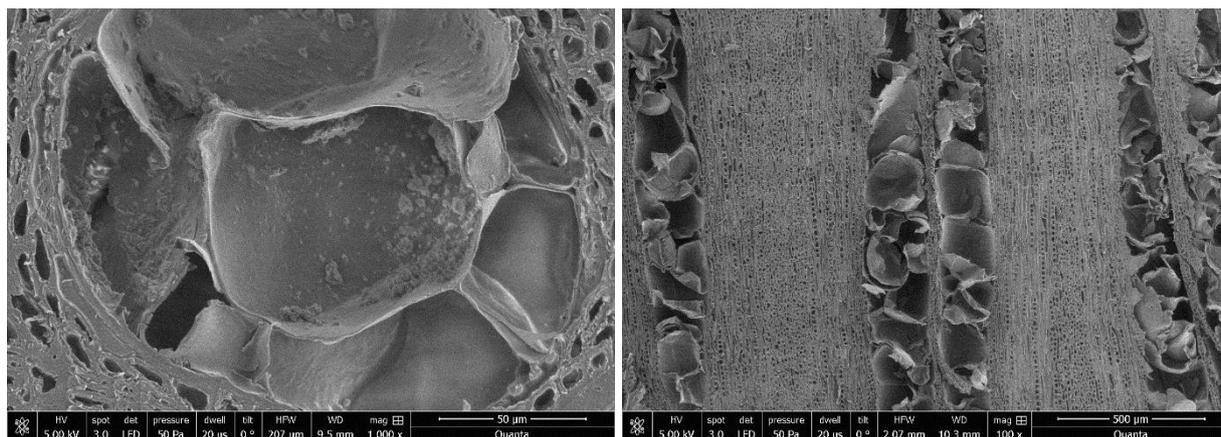
The envelope density of oven-dry wood was determined with GeoPyc 1365 (Micromeritics, Germany). The envelope density was measured on nine oven dried extracted and nine non-extracted replicates of wood specimens ( $10 \times 10 \times 24 \text{ mm}^3$ ). The sample envelope volume is calculated from the difference in volume between the two measurements. For performing envelope density determination of the wood samples, a chamber with 12.7 mm internal diameter was chosen. The skeleton density of the oven dried samples was determined on parallel specimens to avoid possible interferences between the methods. Extracted and non-extracted samples were milled (Ika, Tube Mill 100 control) and pressed into pellets ( $2r = 33 \text{ mm}$ ;  $h = 33 \text{ mm}$ ) in a Chemplex Spectro pellet press (Chemplex Industries Inc., USA) to fit a helium pycnometer AcuPyc 1340 (Micromeritics, Germany) 10  $\text{cm}^3$  measurement chamber.

The decay test was performed according to modified CEN/TS 15083-1 (CEN 2005) procedure. Prior to fungal exposure, samples were oven-dried ( $103 \pm 2 \text{ }^\circ\text{C}$ , 24 h) and their mass was determined. Conditioned samples (65%;  $23 \text{ }^\circ\text{C}$ ) were steam-sterilised prior to exposure to white-rot fungi; 350 mL experimental glass jars with aluminium covers and a hole filled with cotton wool allowing gas exchange with 50 mL of 4 % potato dextrose agar (DIFCO) were prepared and inoculated with *Trametes versicolor* (L.) Lloyd (ZIM L057) and *Hypoxyylon fragiforme* (Pers.) J. Kickx f. (ZIM L108). One week after inoculation, two wood samples per jar were positioned on an HDPE mesh. The assembled experimental glass jars were then incubated at  $25 \text{ }^\circ\text{C}$  and 85 % relative humidity (RH). After incubation, samples were cleaned of adhering surface fungal mycelium, and the mass loss was determined gravimetrically after drying the samples at  $103 \pm 2 \text{ }^\circ\text{C}$  for 24 h. Durability classes (DC) were derived from median mass loss (MLF).

Various moisture performance tests were performed. A comprehensive approach is necessary to address the overall moisture performance of wood. A short-term capillary water uptake test was performed under laboratory conditions ( $T = 23 \text{ }^\circ\text{C}$ ;  $\text{RH} = 50 \%$ ) on a Tensiometer K100MK2 (Krüss, Germany), according to modified EN 1609 standard (CEN 2013b). Long-term water uptake was assessed based on EN 1250-2 standard (CEN 2004). Before the test, specimens were oven-dried for 48 h ( $60 \text{ }^\circ\text{C}$ ) until constant mass, and weighed. In addition to capillary water uptake, wood also absorbs water from the air. An experiment was therefore performed to determine water vapour uptake in a climate with high relative humidity. Specimens were oven-dried for 48 h ( $60 \text{ }^\circ\text{C}$ ) and weighed. The specimens were stacked in a climate chamber with 98–100 % relative humidity. After 24 h of conditioning, their masses were determined again, and their moisture content was calculated. Specimens were then conditioned in the same chamber for an additional three weeks until a constant mass was reached.

## RESULTS AND DISCUSSION

The ring-porous wood of pubescent oak is composed of earlywood and latewood parts, with considerable differences in their structures (Schweingruber et al. 1990). Large earlywood vessels with mean diameters between 200 and 300  $\mu\text{m}$  are formed in spring and are specialised for water transport (Lavrič et al. 2017). A typical feature of earlywood vessels in older growth rings is tyloses, outgrowths from adjacent ray or axial parenchyma cells through pits in vessel walls, which partially or completely block vessels' lumina (Figure 1) (White et al. 1991). In pubescent oak, the first tyloses in solitary earlywood vessels formed in the previous year may appear at the beginning of May, i.e., after a new ring of earlywood vessels is created. In the individual earlywood vessels formed in the current growing season, tyloses are formed in August, just after the cessation of radial growth. The average sapwood width of pubescent oaks is 2–3 cm, i.e. sapwood is composed of 4–6 xylem rings.



**Figure 8: Tangential section of pubescent oak heartwood. Extensive tylosis in the sapwood (left) and heartwood (right) is shown.**

The density of wood is one of the basic parameters that reflect mechanical properties and indicate possible uses. The average oven-dry density of the pubescent oak was 648 kg/m<sup>3</sup>. The density of pubescent oak is similar to the density of European oak (*Q. robur*) (390...650...930 kg/m<sup>3</sup>), American red oak (*Q. rubra*) (480...660...879 kg/m<sup>3</sup>) and Turkey oak (*Q. cerris*) (767 kg/m<sup>3</sup>), while the density of white oak (*Q. alba*) (680...740...785) is a bit higher (Wagenführ 2006). However, there was fairly prominent variability of the density determined, predominantly between sapwood (582 kg/m<sup>3</sup>) and heartwood (681 kg/m<sup>3</sup>) (Table 1). Differences in the density can be explained by the fact that the ring-width of sapwood tissues is wider than that of heartwood. There was no statistically significant difference determined between mature and juvenile heartwood. Extraction resulted in considerable density loss, in the same range as the extractive content. The density of the wood after extraction decreased by 20 % (mature heartwood) up to 25 % (juvenile heartwood) (Table 1). The skeletal density of all wood samples was similar (Table 1), and ranged between 1438 kg/m<sup>3</sup> (mature heartwood) and 1446 kg/m<sup>3</sup> (juvenile heartwood). The skeletal density of beechwood was slightly higher (1456 kg/m<sup>3</sup>). After extraction, skeletal density slightly increased with all of the tissues investigated, which is ascribed to the leaching of extractives with lower density.

In parallel to classic Soxhlet extraction, which enables easier comparison to reference data, extraction with ASE was also performed. Extraction of sapwood and heartwood of pubescent oak in ASE gave an average of 0.52 % (weight/weight based on dry weight) (w/w, dw) and 0.57 % (w/w, dw) of lipophilic extractives, respectively. Significant differences in the content of lipophilic extractives among the wood tissues studied were not found (LSD test). However, the presence of different triterpenes, sterols, triglycerides and fatty acids in the wood of *Quercus* sp. have already been presented in a review by Fengel and Wegener (Fengel and Wegener 2011). The amount of hydrophilic extractives was significantly higher than that of lipophilic extractives. It was measured that sapwood of pubescent oak consists on average of 9.12 % hydrophilic extractives, while the share of hydrophilic extractives in heartwood was found to be much larger, i.e., 15.43 %, as determined by ASE.

A comparative investigation was performed to look at a possible difference in the amount of hydrophilic extractives gained with two polar solvents, i.e., with a mixture of acetone-water (70:30, v/v) and with water only. These two solvents are frequently used for extraction of compounds with a larger molecular mass from plant biomass, e.g., for removal of tannins (Mueller-Harvey 2001). The results of HPLC analysis showed that extraction with water gave the largest amounts of gallic acid from pubescent oak heartwood (Table 1). In contrast, acetone extracts of pubescent oak sapwood and heartwood contained higher amounts of hydroxybenzoic acid than did water extracts

The HPLC analysis showed the presence of gallic acid (GAc, *tr* = 3.2 min), catechin (Cat, *tr* = 6.5 min), hydroxybenzoic acid (HBAc, *tr* = 6.8 min), coumaric acid (CAC, *tr* = 9.4 min) and ferulic acid (FAC, *tr* = 9.9) in wood of pubescent oak. A more substantial amount of gallic acid in extracts of pubescent oak heartwood can also be understood as depolymerisation products of hydrolysable tannins, since the extraction was done at higher temperatures and pressure. However, tannins alone were not quantified in this investigation. Higher contents of free gallic acid can have a decisive influence on wood durability

against wood-decaying fungi. Gallic acid is known to be a natural compound with a potential antioxidant effect. As such, gallic acid could protect wood cells of pubescent oak against free radicals produced by wood-decaying fungi (Belt et al. 2017). Catechin has also been reported to be an excellent free radical scavenger (Choi et al. 2001) and compound with antifungal properties (Yen et al. 2008) and could therefore contribute to the results of the decay test.

**Table 1: Basic Properties of pubescent oak wood. Different letters indicate a statistically significant difference ( $p > 0.05$ ) between different materials tested. EXT marks extracted specimens.**

		SW		MHW		JHW		Beech	
		Avg.	St. dev.						
<b>Density</b>	(kg/m <sup>3</sup> )	582 <sup>a</sup>	53	669 <sup>b</sup>	59	694 <sup>b</sup>	15	647	7
<b>Density EXT</b>	(kg/m <sup>3</sup> )	447 <sup>a</sup>	37	539 <sup>b</sup>	36	515 <sup>b</sup>	29	/	/
<b>Skeleton density</b>	(kg/m <sup>3</sup> )	1440 <sup>a</sup>	8	1438 <sup>a</sup>	8	1446 <sup>a</sup>	3	1456 <sup>b</sup>	2
<b>Skeleton dens. EXT</b>	(kg/m <sup>3</sup> )	1445 <sup>a</sup>	7	1446 <sup>a</sup>	4	1459 <sup>a</sup>	7	/	/
<b>Ext. content</b>	(%)	21.2 <sup>a</sup>	2.8	23.0 <sup>a</sup>	2.2	24.6 <sup>a</sup>	1.7	/	/
<b>Wood Decay Fungi</b>									
<b>Mass Loss (%)</b>	<i>H. fragiforme</i>	28.5 <sup>b</sup>	6.9	0.7 <sup>c</sup>	0.5	0.8 <sup>c</sup>	0.3	35.0 <sup>a</sup>	6.2
	<i>T. versicolor</i>	22.0 <sup>a</sup>	4.7	0.6 <sup>b</sup>	0.5	0.8 <sup>b</sup>	0.3	25.4 <sup>a</sup>	7.9
<b>Durability class</b>		5		1		1		5	
<b>Short-Term Water Uptake (g/cm<sup>2</sup>)</b>	50 s	0.096 <sup>a</sup>	0.013	0.092 <sup>a</sup>	0.010	0.091 <sup>a</sup>	0.015	0.110 <sup>a</sup>	0.010
	100 s	0.108 <sup>b</sup>	0.015	0.101 <sup>c</sup>	0.012	0.102 <sup>c</sup>	0.016	0.147 <sup>a</sup>	0.023
	200 s	0.121 <sup>b</sup>	0.017	0.109 <sup>c</sup>	0.012	0.111 <sup>c</sup>	0.017	0.202 <sup>a</sup>	0.028
<b>Time of immersion</b>									
<b>Long-term Water Uptake (%)</b>	1 h	12.0 <sup>b</sup>	3.0	4.5 <sup>c</sup>	0.4	5.0 <sup>c</sup>	1.0	31.2 <sup>a</sup>	5.4
	24 h	40.4 <sup>b</sup>	6.3	15.3 <sup>c</sup>	1.2	16.9 <sup>c</sup>	2.5	65.4 <sup>a</sup>	4.3
<b>Time of condition.</b>									
<b>Water Vapour Uptake (%)</b>	24 h	10.3 <sup>b</sup>	0.9	5.7 <sup>c</sup>	0.4	6.2 <sup>c</sup>	0.5	11.6 <sup>a</sup>	0.3
	4 w	30.6 <sup>a</sup>	2.8	22.8 <sup>c</sup>	0.5	22.8 <sup>c</sup>	0.5	25.0 <sup>b</sup>	0.6

White rot fungi are the predominant cause of hardwood degradation in outdoor applications (Schmidt 2006) so only white-rot fungi were used in this research. *Trametes versicolor* and *Hypoxylon fragiforme* are fungi that are also typically found on oak wood (Schmidt 2006). Mass loss of the reference beechwood was between 35.0 % (*H. fragiforme*) and 25.4 % (*T. versicolor*), which indicates that the fungi were vital and capable of wood degradation. Sapwood proved to have durability in the same range (Table 1) (EN 350 2017). Mass loss of sapwood was slightly lower, between 28.5 % (*H. fragiforme*) and 22.0 % (*T. versicolor*). According to standard CEN/TS 15083-1 (CEN 2005), sapwood can be classified into durability class (DC) 5. In contrast to sapwood, heartwood was much more durable. None of the samples exposed to the wood decay fungi lost more than 2.0 % of the mass, in spite of a rather narrow ring width (around 1 mm). European oak (*Q. robur*), with such narrow rings, has the poorest durability (Humar et al. 2008). In spite of the narrow ring width, pubescent heartwood can be classified into the group of most durable wood species (DC 1) according to CEN/TS 15083-1 (CEN 2005) procedure. There was no statistically significant difference observed between mature and juvenile heartwood.

In addition to inherent durability, which is the result of the presence of toxic secondary metabolites and/or biocides in wood, water performance has been identified as another important factor influencing the performance of wood in outdoor applications (Meyer-Veltrup et al. 2017). Wood with better water exclusion efficacy lasts longer than similar wood with lower water exclusion efficacy, regardless of similar inherent durability. Short-term water uptake can be used as an indicator for water performance in the axial direction. Axial planes are usually the weak point for fungal infestation, which makes this indicator very important (Žlahtič and Humar 2016). Higher short-term uptakes were determined with sapwood (0.121 g/cm<sup>2</sup>) than with heartwood (0.110 g/cm<sup>2</sup>) (Table 1). These values are similar to the values determined on European oak (0.110 g/cm<sup>2</sup>) (Žlahtič and Humar 2016). However, these values are considerably lower than values determined with beechwood (0.202 g/cm<sup>2</sup>). One of the reasons for the differences can be ascribed to tyloses, which can be seen in sapwood (Figure 1) and even more extensively in heartwood.

In addition to short-term water uptake, long-term water uptake was also determined. These measurements comprehensively indicate water performance, since all axial planes are exposed to water. Similarly, as

indicated by capillary water uptake, pubescent oak wood performed much better than the reference beechwood. For example, after one hour of immersion (MC1h), pubescent oak wood sapwood samples reached 12.0 % moisture content, and heartwood samples uptake significantly lower amounts of water (MC1h 4.5 % to 5.0 %). Moisture uptake with beechwood samples was much higher (MC1h 31.2 %) (Table 1). If long-term water uptake of pubescent oak is compared to the moisture performance of European oak heartwood (MC1h 16.9 %), it can be seen that pubescent oak exhibits much better moisture performance than European oak. This can be ascribed to chemical and anatomical differences between the wood species. Two indicators in this study characterise sorption properties. Firstly, the moisture content of the wood after one day of conditioning in a chamber with 98 % – 100 % relative humidity was determined. The moisture content of pubescent oak sapwood (10.3 %) was slightly lower than the moisture content of reference beechwood (11.6 %). However, if all moisture tests are summarised, it can be concluded that the moisture performance of pubescent oak heartwood is much better than that of reference beechwood, and better than the moisture performance of European oak determined in previous studies.

## CONCLUSIONS

The ring-porous wood of pubescent oak was composed of earlywood and latewood parts with considerable differences in their structure. Large earlywood vessels with mean diameters between 200 and 300 µm contain characteristic tyloses, which partially or entirely block vessels' lumina.

The average oven-dry density of the pubescent oak was 648 kg/m<sup>3</sup>, which was similar to the density of European oak (*Q. robur*), American red oak (*Q. rubra*) and Turkey oak (*Q. cerris*). However, there was fairly prominent variability of the density determined, predominantly between sapwood and heartwood. Pubescent oak has high extractive content. Heartwood was rich in phenolic extractives. Among the identified compounds, gallic acid was assigned to the most abundant chromatographic peak.

The durability of the oak wood sapwood was similar to the durability of beech, while heartwood was classified in the group of most durable wood species (DC 1). Excellent durability can be ascribed to the presence of biologically active extractives. In parallel to durability, oak heartwood was characterised by excellent water performance, which was a consequence of the anatomical composition, predominately the presence of tylosis.

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## Wood composites vs mycelium composites – water absorption and mould growth properties

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**Keywords:** building materials; moulds; mycelium composites; volumetric swelling; water absorption; wood composites

### ABSTRACT

In this study, the water absorption properties and mould growth of commercial wood composites (WC) and mycelium based composites (MC) were compared. The following WC materials were selected: cemented wood wool (CW), oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture resistant particle board (MRPB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF), moisture resistant medium density fibreboard (MRMDF). The MC were fabricated in the laboratory from agricultural (hemp mycelium composite; HMC) and forestry (birch wood mycelium composite; WMC) by-products which were bounded together with the natural growth of the fungal mycelium. In the WC group, the lowest water absorption (below ~30%) after 24 h was observed for OSB, MDF, LMDF and MRMDF. The LPB had significantly higher absorption (~85%) than PB (~47%), while MRPB had lower absorption (~35%) compared to PB. The WC made of larger size wood particles like OPP, OSB and BP had 43, 28 and 35% water absorption after 24 h, respectively. The MC group had the highest water absorption from all tested materials, reaching 400-550% after 24 h. The volumetric swelling of all tested specimens was below 25%. Very low swelling (under 5%) was observed for MC despite their highest water absorption. The mould growth on WC materials was not observed after 2 days exposure; then it gradually increased and the most pronounced growth (3.5-4) after 14 days occurred on OSB, BP, PB, MRPB, MDF and MRMDF. MC materials were totally covered by moulds already after 2 days exposure, showing that the initial hours/ days after material's moistening are critical regarding the mould development. The fungal microflora on material surfaces consisted of the genera *Trichoderma*, *Penicillium*, *Acremonium* and *Rhizopus*.

### INTRODUCTION

Water penetration into building materials in a house is often due to water leakage from pipes, groundwater penetration, condensation, or damp air. When water intrusion occurs, one critical factor that affects water penetration into building materials is the water absorption rate of the materials (TenWolde and Rose 1993). Mould growth on the surfaces of various materials is common in damp houses; the best method to control mould growth is to prevent water from entering the materials (Zabel and Morrell 1992). Many indoor moulds produce very potent mycotoxins when growing on building materials (Nielsen 2003) and it is possible that these toxins may play an important role in the adverse health effects observed in mould infested buildings.

For mould development, the critical relative humidity (RH) requirement is between 80% and 95% depending on other factors such as ambient temperature, exposure time and the type and surface conditions of building materials (Viitanen et al. 2010). The susceptibility of different building materials (wood-based materials, gypsum boards and inorganic boards) to mould growth varies. Some are tolerant to the high RH in ambient air without occurring mould growth, while others are less tolerant, and mould can grow at RH as low as 75% (Johansson et al. 2012). It has been found (Pasanen et al. 1991) that the repeated or persistent moisture condensation or water leakage is sufficient for fungal germination and growth on building

materials even at low RH. A study on the water absorption rates of different building materials shows (Yang 2008) that solid wood absorbs the least quantity of water, followed by plywood and wood composite panels with the highest water uptake.

There are a wide variety of wood composite (WC) products that can be made from wood. Sawdust, planer shavings and bark have been used to produce composite boards. There are also a lot of WC that are a combination of wood and non-wood elements. Combinations of wood and inorganics and synthetic polymers have been produced; some are commercial, and some are still in the research phase (Rowell 2005). Mycelium based composites (MC) are an emerging category of biocomposites relying on the valorisation of lignocellulosic wastes and the natural growth of the living fungal organism. While growing, the fungus cements the substrate, which is partially replaced by the tenacious biomass of the fungus itself. The final product can be shaped to produce insulating panels, packaging materials, bricks, or new-design objects. MC show low density and good insulation properties, both related to acoustic and thermal aspects (Girometta et al. 2019).

This study was conducted to determine the water absorption rates and thickness swelling of various commercial WC and our novel MC after water immersion and the corresponding mould growth on the non-sterile surfaces.

## EXPERIMENTAL METHODS

### *Materials*

Different WC materials were selected: cemented wood wool (CW), oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture resistant particle board (MR PB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF), and moisture resistant medium density fibreboard (MRMDF).

The MC were manufactured in our laboratory according to Zimele et al. (2020) from agricultural (hemp mycelium composite, HMC) and forestry (birch wood mycelium composite, WMC) by-products which were bounded together with natural growth of fungal mycelium.

### *Water Absorption and Volumetric Swelling*

The water absorption and thickness swelling of WC and MC materials were measured according to ASTM D1037: 2012, using 5 cm x 5 cm specimens for wood based building materials and 3 cm × 3 cm specimens for mycelium based materials (6 replicates were used in each group). The specimens were immersed in distilled water and the weights were measured after 2 h and 24 h and volumes after 24 h. The water absorption and volumetric swelling values were determined from the weight and volume difference in relation to the initial weight and volume.

### *Mould Growth*

After the water absorption test, the moistened specimens were subjected to the natural mould contamination from indoor air. The experiment was performed in cultivation chambers at 22°C and 75% RH for 14 days, with a periodical assessment of fungal growth using the rating scale 0–4: 0 – clean, 0% attack; 1 – trace, 5% growth; 2 – slight, 6–25% growth; 3 – medium, 26–50% growth; 4 – severe, > 50% growth. More contaminated surface of each specimen was assessed excluding edges.

### *Light Microscopy*

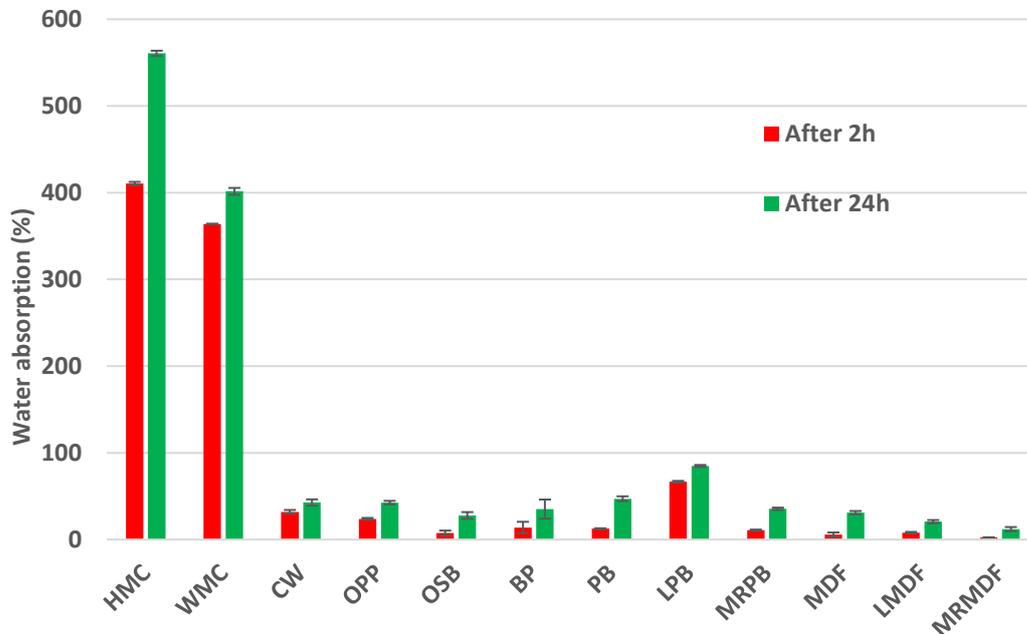
The fungal samples were stained with *lactophenol blue* solution (Fluka) and identified with a light microscope *Leica DMLB* at magnifications of ×50, ×100, ×200 and ×400. The images were captured with a video camera *Leica DFC490* using calibrated image analysis software (Image-Pro plus 6.3, Media Cybernetics, Inc.).

## RESULTS AND DISCUSSION

### *Water Absorption and Volumetric Swelling*

The water absorption and swelling are important properties for the application of WC and MC in building materials or insulation elements, and they characterise the hydrophilicity of the materials.

Fig. 1 shows the water absorption results of all tested specimens. HMC and WMC had the highest water absorption, reaching 400-550% after 24 h. Water absorption in similar research of hybrid panel composites based on wood, fungal mycelium and cellulose nanofibrils after 24 h of the immersion period was 120-240% (Sun et al. 2019). Probably, wood particles in WMC and hemp in HMC were too hydrophilic and needed additional treatment before the preparation of the composites to limit water absorption.



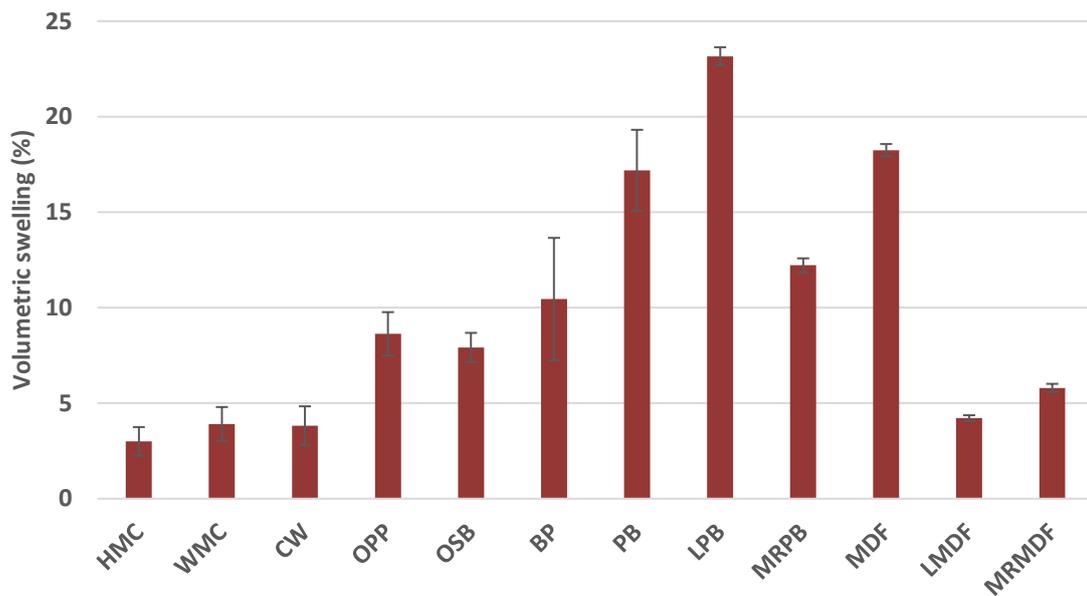
**Figure 1: Water absorption after 2 h and 24 h for the tested specimens - hemp mycelium composite (HMC), wood mycelium composite (WMC), cemented wood wool (CW), oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture resistant particle board (MRPB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF), moisture resistant medium density fibreboard (MRMDF)**

CW specimens showed no significant difference in water absorption after 2 h and 24 h that was in a range of 30-40%. The lowest water absorption (below ~30%) after 24 h was observed for OSB, MDF, LMDF and MRMDF. Laminate on particle board did not protect from water and LPB had significantly higher absorption (~85%) than PB (~47%), while the moisture resistant particle board MR PB had lower absorption (~35%) compared to PB. In similar research, the physical-mechanical properties of particle boards manufactured with Eucalyptus wood, bamboo and rice husk particles were assessed. Water absorption of wood particle board was ~43%, and for other combinations, 67-72% (Melo et al. 2014)

Untreated and uncoated MDF had the highest water absorption (~31%) compared to other MDF specimens. Laminated MDF had significantly lower (~21%) and moisture resistant MDF had the lowest (~12%) water absorption. This confirmed that, in the case of MRPB and MDF, commercial treatment against water absorption is effective. For MDF manufactured using bamboo, the water absorption after 24 h was 15-28% for different specimens (Marinho et al. 2013).

Wood products made of larger size wood pieces like OPP, OSB and BP had the water absorption of 43%, 28% and 35% after 24 h, respectively.

The volumetric swelling of all tested specimens was below 25% (Fig. 2). Surprisingly, very low swelling (under 5%) was observed for the mycelium composites HMC and WMC despite their highest water absorption (400-550%) from all tested specimens. This indicates that the increased water absorption of HMC and WMC affected the material's dimensions negligibly. This can be attributed to the low density of these specimens, and a large surface for water absorption was available in material pores. Probably, water related properties depend on the composite manufacturing methodology, because the thickness swelling of hybrid panel composites based on wood, fungal mycelium, and cellulose nanofibrils after 24 h can reach up to 50-130% (Sun et al. 2019).



**Figure 2: Volumetric swelling after 24 h immersion in water for the tested specimens - hemp mycelium composite (HMC), wood mycelium composites (WMC), cemented wood wool (CW), oak-pine shield parquet (OPP), oriented strand board (OSB), birch plywood (BP), particle board (PB), laminated particle board (LPB), moisture resistant particle board (MRPB), medium density fibreboard (MDF), laminated medium density fibreboard (LMDF), moisture resistant medium density fibreboard (MRMDF)**

The lowest swelling was observed for CW, probably, due to the use of cement for fabrication of this material. The highest swelling (over 10%) was observed for BP, PB, LPB, MRPB and MDF. All particle board specimens had high swelling. This is, probably, because small size wood particles are used for manufacturing of these composites and they easily swell after water absorbance. Laminate on particle board did not protect from water absorption and swelling for LPB was significantly higher (~23%) than for PB (~17%). Moisture resistant PB demonstrated the lowest swelling (~12%) compared to other tested particle boards. This indicates that the treatment for the limitation of water absorption is effective. For particle boards manufactured with Eucalyptus wood, bamboo and rice husk particles, the thickness swelling was 31%, 30% and 49%, respectively (Melo et al. 2014).

MDF had the highest swelling (~18%) compared to laminated MDF (~4%) and moisture resistant MDF (~6%). Unlike particle board, the laminate on MDF protected from water absorption and, unexpectedly, swelling for laminated MDF was even lower compared to that of moisture resistant MDF. For MDF manufactured using bamboo, swelling was 11-21% for different specimens (Marinho et al. 2013).

The swelling of wood products made of larger size wood pieces like OPP, OSB and BP was similar (~8-10%) in terms of measurement error limits.

### **Mould Growth**

The accelerated method ensured the natural contamination with common mould fungi from the indoor environment. Water soaked specimens served as model samples of wet building materials in the case of water leakage, condensation or other moisture influenced damage that occurs in buildings.

The mould growth on wet WC materials was not observed after 2 days exposure (Table 1). After 6 days, the most noticeable mould growth (rating 3-4) was observed on PB, MRPB and MDF specimens soaked with water for 24 h. The water absorption for these materials was from 31% to 47% (Fig. 1). The moulds gradually spread also on other materials, and after 14 days exposure, severe fungal growth occurred on OSB, BP (rating 4) and MRMDF (rating 3.5). The water absorption for these materials ranged from 12% (MRMDF) to 35% (BP) (Fig. 1). Surprisingly, the moisture resistant MDF was also susceptible to mould contamination.

**Table 1: Mould growth on 2 h and 24 h water soaked WC and MC materials during 14 days exposure**

Material	Time [days]									
	2		6		9		12		14	
	2 h	24 h	2 h	24 h	2 h	24 h	2 h	24 h	2 h	24 h
HMC	4	4	4	4	4	4	4	4	4	4
WMC	4	4	4	4	4	4	4	4	4	4
CW	0	0	0	0	0	0	0	0	0	0
OPP	0	0	0	0	0	0	0	0	0	0
OSB	0	0	2	2	2	2	2.5	3	3.5	4
BP	0	0	0.5	1	1.5	4	2	4	2	4
PB	0	0	0	3.5	0.5	4	1	4	1	4
LPB	0	0	0	0	0	0	0	0	0	0
MRPB	0	0	0	4	0	4	0	4	1	4
MDF	0	0	0	3	0	4	0	4	1	4
LMDF	0	0	0	0	0	0	0	0	0	0
MRMDF	0	0	0	1	0	2.5	0	2.5	0	3.5

Table 1 shows that the incubation conditions 22°C/ 75% RH were acceptable for mould development on some WC materials in the first exposure days. Nielsen et al. (2004) reported that the lower limit for mould growth on conditioned wood and wood composites was 78% RH at 20-25°C after more than 15 weeks. This shows a noticeable difference in the mould infestation speed over moistened and conditioned (dry) materials at similar climate conditions. Consequently, a quick drying of wet building materials in the first days plays a crucial role to avoid mould development.

Contamination was not observed on CW, LPB, LMDF and OPP materials. This is explained with a specific structure of materials. CW contains cement that could (i) protect wooden fibres and (ii) increase alkalinity. Both factors could affect the mould growth. The laminated surfaces of PB and MDF served as a protective layer of the inner board material. We observed the mould growth on narrow, non-laminated edges of LPB and LMDF because water penetrated and moistened the inner part. The top surface of OPP was free of contamination because of the surface treatment with a protective coating. On the reverse, non-treated surface and narrow edges, a trace of moulds was observed.

As expected, the water absorption time affected the mould distribution on WC. The 2 h water absorption caused negligible mould growth (rating 1-2) (except for OSB) on more susceptible materials while 24 h were critical for most of the materials with serious mould development (rating 3-4).

MC materials were totally covered by moulds already after 2 days exposure, showing that the initial hours (2-24 h) after the materials' moistening are critical for mould development (Table 1). The water absorption of both HMC and WMC materials also reached the highest values (Fig. 1). This stimulated the immediate contamination by *Rhizopus* followed by *Trichoderma* species. To improve the mould resistance of MC, the water absorption properties should be reduced by the impregnation or surface treatment with a hydrophobic substance. The fungal microflora on WC and MC material surfaces consisted of the genera *Trichoderma*, *Penicillium*, *Acremonium* and *Rhizopus*. Similar moulds were identified by Ahmed et al. (2013) on conditioned Scots pine sapwood samples exposed to different temperature (> 20°C) and RH (> 90%) levels. After natural contamination, severe mould infestation was observed after 12-14 days incubation (Ahmed et al. 2013).

## CONCLUSIONS

The water absorption properties between WC and MC considerably differed: MC had significantly higher water absorption levels. In contrast, the volumetric swelling of MC was lower than that for most of the WC materials. This is attributed to the low density of MC and the large surface in material pores for water absorption.

There was a considerable difference between WC and MC regarding mould growth: in the first exposure days, severe mould growth occurred on MC materials while WC products were initially resistant against

moulds, and contamination progressed in the later days. The first 2 days were critical to avoid moulds on wet WC materials. In this period, efficient drying could protect these materials against the mould attack. The water absorption time affected the mould distribution on WC. The 2 h water absorption caused negligible mould growth while 24 h were critical for most of the WC materials. The most susceptible WC materials to mould infestation were PB, MR PB and MDF but the most resistant ones were CW, LPB, LMDF and OPP. MC materials were not resistant against moulds. The drying of MC immediately after water soaking could stop the contamination. To increase the water and mould resistance of MC materials, they should be treated or covered with a waterproof substance to improve hydrophobic properties.

## ACKNOWLEDGEMENTS

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## Impregnation of 'Pannonia' poplar with paraffin

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**Keywords:** 'Pannonia', poplar, density, paraffin, impregnation

### ABSTRACT

Timber used for pencil production must meet rigorous quality requirements, in order to achieve satisfactory finished product quality. The impregnation of poplars as potential raw materials with paraffin wax may help in reaching this quality level. The 'Pannonia' poplar is one of the hybrid poplars with higher densities. Its impregnability with paraffin is greatly influenced by its density. The emulsion uptake of a species decreases as its density increases. The material examined had an air-dried density of 0.39-0.48 g/cm<sup>3</sup>, which could take up to 140-200% of emulsion, with respect to its initial mass. The 3 impregnation processes, using different methods, have produced identical emulsion uptake results. There was no difference between the impregnation schedules in terms of paraffin uptake.

### INTRODUCTION

The high demand for wood species and decline in juniper production has contributed to the interest in an alternative raw material for use in the pencil production industry (Kaygin et al. 2015). The highest quality of wood for pencils and colour pencils is produced by the Californian cedar and the Brazilian pine. The wood used in manufacture should be as knotfree, long-fibred and uniformly grown as possible in order to ensure easy and neat sharpening of the manufactured pencil. Poplars are also present among the species that can be potentially used for pencil production, and their rapid growth may help them supplement or even replace the currently used species.

The natural lifestyle of poplars is of a pioneering nature: they prefer raw soils and fresh washlands where they are capable of rapid growth and high timber yields. These properties prompted growers to breed economically valuable cultivars. The utilisation possibilities of hybrid poplars are identical to indigenous species, but they have the advantage of potential improvement possibilities of their properties through genetic modification (Rathke et. al 2012). They are not in widespread use as construction timber, due to their low strength properties. Their broader utilisation calls for the improvement of their relevant physical and mechanical properties (Wang et al. 2015).

In Hungary, currently 18 poplar cultivars are recognised by the state and registered in the national cultivar registry for forestry purposes. The cultivar 'Pannonia' is one of the most significant in Hungarian poplar breed cultivation, being second among all poplars in terms of total cultivation area. Its juvenile growth is especially dynamic, approaching that of 'I-214' in this stage. In spite of the lower average age of Hungarian populations, it has a larger average diameter than other poplars found in Hungary. Its capability to successfully adapt to a broad range of growth zones, rapid and dynamic growth, high timber yield and resistance to diseases makes it a specially significant plantation cultivar.

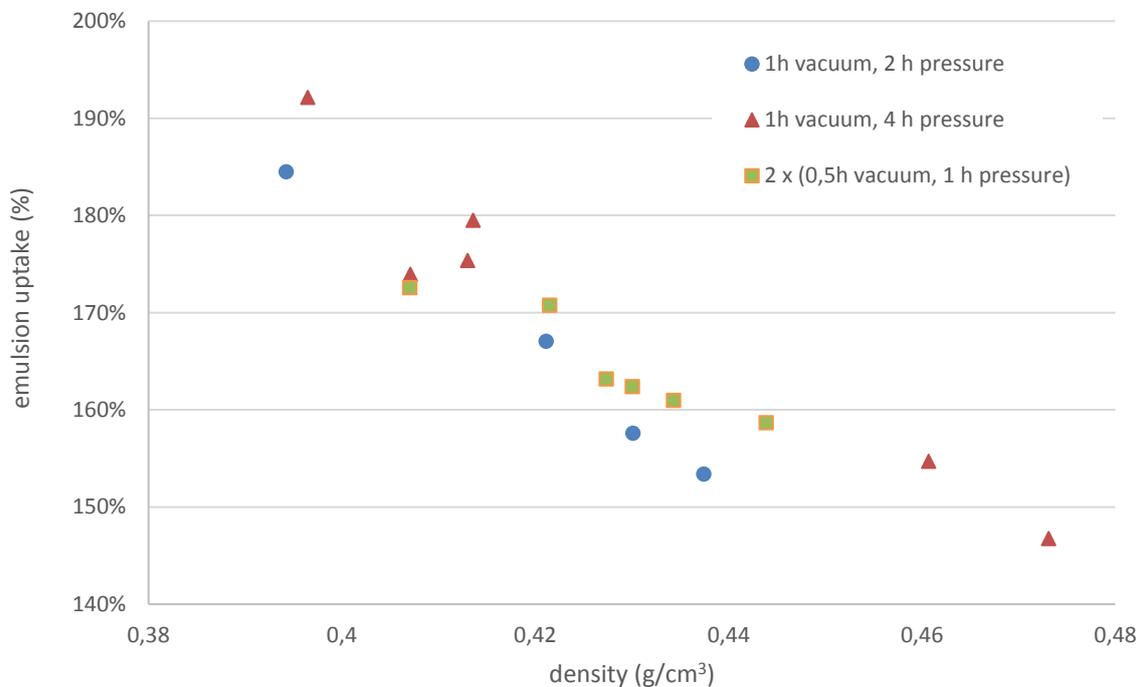
Water repellent treatments could increase water repellency and dimensional stability of wood, as well as reduce wood checking increased by outdoor weathering. The most common water repellent applied to the wood industry is paraffin wax (Schultz et al. 2007). There are different penetration patterns among species owing to their different anatomical features (Zhang et al. 2006). In the pencil production technology, impregnation of the raw material with paraffin may become necessary to improve the finished product's sharpening properties and its general suitability for the end purpose.

## EXPERIMENTAL METHODS

The experimental samples were 4x8x20 (radial×tangential×longitudinal) cm in size and had 7-7,5% moisture content (dry basis). The aqueous paraffin emulsion was painted, to visualize the penetration depth. Treatments were performed with 3 different methods, but with the same vacuum and pressure values. Changed parameters were in the duration of vacuum and pressure used. Prior to treatments, the density of the test pieces was determined by mass and volume measurement.

## RESULTS AND DISCUSSION

Density of examined samples were between 0,39-0,48 g/cm<sup>3</sup>, which is the same as in the various literature (Molnár, Komán 2006). The lower the initial density, the higher the emulsion uptake. The results show, that the material, the density of which is below 0,4 g/cm<sup>3</sup> can uptake nearly two times of its original weight, while in the case of materials, the density of which is above 0,44 g/cm<sup>3</sup> can uptake nearly 1,5 times of its original weight. The applied treatment periods were of no relevance in this respect.



*Figure 1: The relationship between the density and emulsion uptake*

The test results show, that regardless of the treatment methods employed, the density has a clear influence on the amount of emulsion uptake. The correlation values are very high for all three treatments (Table 1.). More precise results may be available upon impregnations performed on a greater number of samples.

*Table 1: Correlation coefficients between density and emulsion uptake*

treatment method	correlation coefficient
1h vacuum, 2 h pressure	0,96
1h vacuum, 4 h pressure	0,93
2x (0,5h vacuum, 1 h pressure)	0,88

## CONCLUSIONS

The impregnability of 'Pannonia' poplar with paraffin is greatly determined by the timber's density. The samples with an air-dried density of 0.39-0.48 g/cm<sup>3</sup> can take up to 140-200% of the emulsion, with respect to their initial masses. The uptake volume decreases proportionally to the increase of density. The 3 impregnation methods applied have produced identical emulsion uptake results. The influence of density could be equally demonstrated in all three processes. There are no differences among the impregnation processes in terms of emulsion uptake. From the perspective of the even impregnation of the finished product across its cross section, control based on weighing may be an important parameter.

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## Ash content of poplar species

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**Keywords:** poplar, species, ash content, wood, bark

### ABSTRACT

Nowadays, the role of biologically renewable energy sources is gaining increased importance within the energy sector. In this field, the various hybrid poplar species are in keen use, generating significant volumes of ash. This is important from the perspective of the influence on technological processes and the calorific value. As is the general case concerning the various wood species, the wood of the poplars involved in the experiment ('Kornik', 'I-214', 'Pannonia', 'Raspalje', 'Koltay') also show a significantly lower ash content (1,15 – 2,31%) than the bark (4,41 – 6,92%). For wood, the value of 'I-214' is smallest, while 'Raspalje' is greatest. For the bark, the ash content of the 'Koltay' clone is far more favourable than those of the other 4 clones, which are in the same order of magnitude. The difference between the wood and the bark within a species is most favourable in case of 'Koltay', where the ash content of the bark is 2,7 greater than that of the wood. From this perspective, the 5,3-fold difference of 'I-214' is the most unfavourable.

### INTRODUCTION

The present-day increasing energy demand and the uncertainties that may occur in energy supply have focused the attention to CO-neutral and sustainable energy sources. Application of biomass for energy purposes has become more and more common and has increased the significance of wood heating (Füzesi 2014). The majority of biomass is formed by dendromass, i.e. the wood based biomass. This is mainly explained by the fact that wood is an easily treated energy carrier with minimal sulfur and low ash content, its calorific value close to that of brown coal, and its combustion only generates so much CO<sub>2</sub> the tree had bound from the atmosphere during its growth – it is therefore an environmentally friendly energy carrier (Vágvölgyi 2014).

The use of various biomasses for energy is significantly determined by their calorific values, ash contents and other combustion parameters. The energy parameters of various wood species are however determined by their genetic properties, structure, macroscopic parameters and the age of the population in question. Wood as a fuel has four definitive parameters that determine its suitability for energy uses: density, calorific value, moisture content and ash content and the constituents of these.

Naturally, the greater utilization of biomass energy is accompanied with the increase in the amount of combustion by-products, that is with the increase in the amount of ashes. The properties of wood ash depend on a number of factors, among others on the plant species and the parts of the plant combusted (bark, wood, leaves), their combination with other fuel sources, the soil and climatic conditions, and the circumstances of combustion, collection and storage (Etiégni, Campbell 1991; Someshwar 1996). In comparison to the wood, the bark shows a greater variability with respect to ash content. The wood usually has a lower relative ash content, while that of the bark is significantly higher (Passialis et al. 2008; Nosek et al. 2016).

The noncombustible slag formed during the energy use of biomass raises special operational problems in larger firing plants. This is connected in part to damages of the firing equipment, and in part to the deposition of large volumes of ash. The quantity of ash content is not negligible either, as Lieskovsky et al. claim an increase of 1% of the ash content results in a decrease of 0,11 MJ/kg of heating value. The purpose of the research is to establish the variability of the ash content of the wood and bark of the various hybrid poplar species, taking into account the differences between clones.

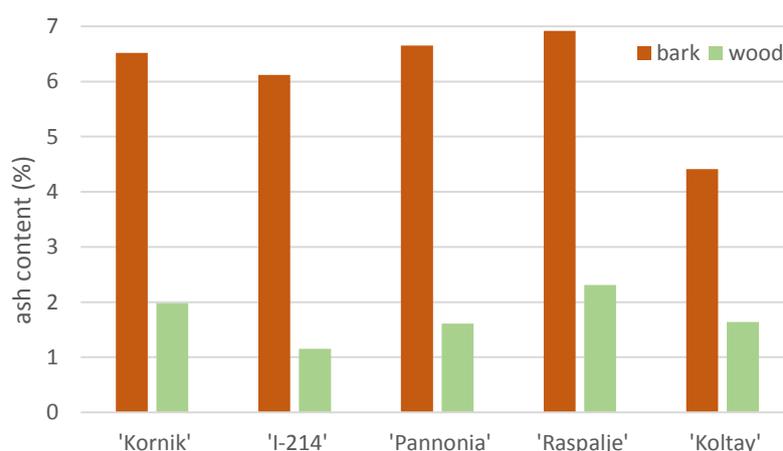
## EXPERIMENTAL METHODS

The hybrid poplars involved in the test were 'I-214', 'Koltay', 'Kornik', 'Pannonia' and 'Raspalje', originating from the same growth zone, all of them aged 28 years. For the tests, 5 cm thick discs cut at the diameter at breast height were used. Ash content test took place on oven-dry samples, separately for the wood and the bark. Following reduction, a 2 g quantity of the samples were analysed according to the ISO 18122:2015 standard. 3 measurements per sample were taken.

## RESULTS AND DISCUSSION

From the perspective of ash content, significant differences are primarily observable between the wood and the bark. The ash content of the bark is significantly higher than that of the wood, as unanimously established by former references (Klašnja et al. 2002; Dzurenda et al. 2014; Hytönen, Nurmi 2015). This value encompasses a narrower range for the wood of the species tested (1,15 - 2,31%), while a broader one for the bark (4,41 – 6,92%). For the wood, somewhat higher values can be observed, as referenced for younger specimens by various studies (Fengel, Wegener 1985; Klašnja et al. 2013; Komán 2018; Vusić et al. 2019).

For the ash content of the bark, the most advantageous value was demonstrated by the 'Koltay' clone (4,41%) exceeded by the figures for the other 4 clones by 38-56% (Figure 1). Concerning the wood, the smallest value was obtained in case of the 'I-214' clone (1,15%), followed by the other clones with a minimum 40% and maximum value of 200%. For the 'I-214' clone, a similar order of magnitude can be observed for the wood as well as the bark, as for a plantation 2 years old (Komán 2018).



*Figure 1: Ash content of wood and bark*

The ash content of the bark and the wood may be significantly different within a species. From this point of view, the 'Koltay' clone is the most favourable, where the value for the bark is 2,7 times greater than that for the wood. The 'I-214' is the most unfavourable, where the difference is 5,3-fold. According to tests conducted by Vusic et al. (2019), the difference may even be 10 fold.

## CONCLUSIONS

The statement generally applicable to wood species and hybrid poplars, i.e. that bark has a greater ash content than the wood, is likewise applicable to the various poplar clones. From this aspect, there are significant differences between the clones. For the tested 5 clones, this value is found between 2,7-4,3. The smallest difference can be found in case of the 'Koltay' clone, with the largest in case of the 'I-214' clone. The ash content of the wood for the tested hybrid poplars varies between 1,15 – 2,31%, while that of the bark between 4,41 – 6,92%. The most favourable values

for the test of the wood were obtained for 'I-214', and 'Koltay' produced the best results for bark. The 'Raspalje' clone has the highest ash content for both wood and bark.

### ACKNOWLEDGMENTS

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## Simulation of heat transfer in wood

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**Keywords:** heat transfer, poplar, black locust, norway spruce, beech

### ABSTRACT

During the various technological processes in the timber industry, raw materials must be treated and stored at specified temperatures. In these cases, the heat transfer inside the timber must be known, with the help of which time, energy and cost can be saved, and subsequent problems with the finished product can also be avoided. The processes of heating of poplar, spruce, beech and black locust species due to higher external temperatures were successfully modelled using the finite element method. The difference between the model and the actual temperature varies in the 0,7-3,7 °C range. Density significantly influences heat transfer, as the species with a lower density heated up faster throughout their cross sections. The influence of the anatomical directions was demonstrated in that the longitudinal heating of the timber was faster than the figures corresponding to the radial and tangential directions. There were practically no differences between the two transversal heat transfer.

### INTRODUCTION

Preparation of wood is a delicate process, requiring significant care, prior to the technological processes of the products to be manufactured. In appropriate cases, this starts as early as with the storage, which takes place under controlled temperature and humidity values. Unfortunately, in many cases, these external factors are not controlled, as a result of which various problems emerge during the technological process or in the finished product. Hence the importance of knowing the effect of the change of external temperature on the temperature of the wood. This is particularly justifiable in regions where the temperature differences between the various seasons are significant.

From the perspective of thermal conductivity, the most important factors are species, density, moisture content, direction of heat flow (anisotropy), inclination of grain, and relation of volume or thickness of the sample to moisture content (Suleiman et al. 1999). The study is focused on two of these, namely: species and anisotropy.

The thermal conductivity of porous timber is lower, as the thermal conductivity of the air filling the cavities is lower than that of the cell walls (Rohsenow 1973). For the heat flow, cell walls are an advantage, while the air in the cell cavities blocks it (Cote, Kollmann 1968). Free water conducts more heat than bound water, therefore the increase of the thermal conductivity is greater above the fibre saturation point (Siau 1971).

The simulation of heat transfer within wood samples provides useful insight into the heat transfer mechanisms (Zhang et al. 2017). The thermal properties of wood vary in the 3 anatomical directions. Longitudinally, the thermal conductivity factor is greater than transversally. This is due to the orientation of the molecular chains within the cell wall. The cellulose bundles in the cell walls are organised in so-called microfibrils, mostly oriented parallel to the longitudinal axis of the cell. Obviously, thermal conductivity is greater along the length of the microfibrils than perpendicularly to them (Parrot, Stuckes 1975). Radial conductivity may be greater than the tangential one, thanks to the radially oriented rays. The correlation of the two transversal conductivities is determined by the quantity of rays for deciduous trees and that of latewood for coniferous species (Steinhagen 1977).

The tests do not only offer help preparation of production, but also concerning the behaviour of structures and products placed outdoors or within reach of the external conditions. The industrial players may obtain information, with the help of which they may prevent problems otherwise emerging during production or in the finished product.

## EXPERIMENTAL METHODS

4 different species were involved in the research: beech (*Fagus sylvatica* L.), black locust (*Robinia pseudoacacia* L.), spruce (*Picea abies* (L.) H. Karst.), and poplar (*Populus*) were tested. The measurements were performed using an universal measuring instrument of type Ahlborn Almemo 2590 with the help of a NiCr-Ni thermowire. The cubic test specimens as shown in Figure 1, with edges 46 mm long were insulated all around using polyurethane foam so as to leave the desired anatomical direction free. The samples were put in the conditioning chamber after conditioning at normal climatic conditions. When examining the differences between wood species, the relative humidity in the conditioning chamber was 65% at 60 °C, while the figure corresponding to the anatomical directions was 55% at 50 °C. The measurements were performed in both cases on 3 specimens per wood species.

The finite element method analysis was performed by using the Transient Thermal function of the ANSYS 2019 R2 – Workbench programme.

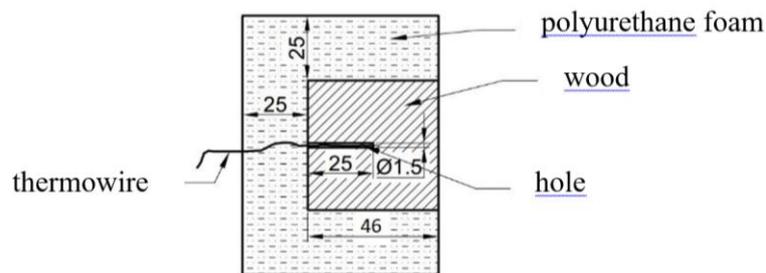


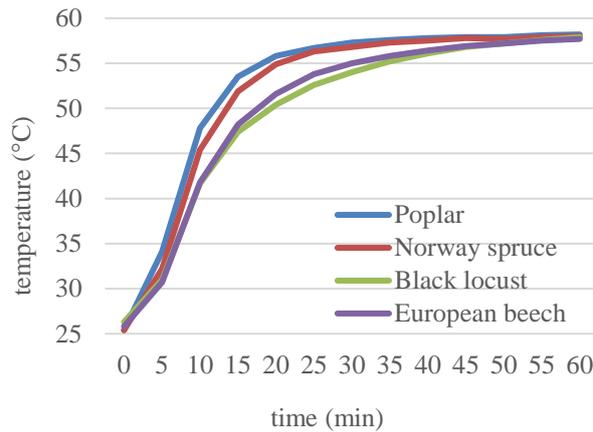
Figure 1: Specimen prepared for tests

## RESULTS AND DISCUSSION

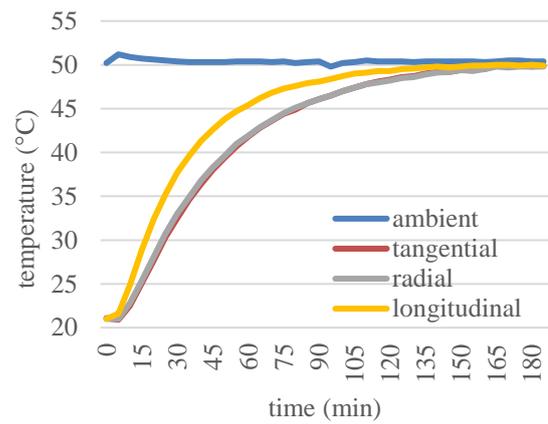
The influence of density is well demonstrated in Figure 2. The densities of the 4 species corresponding to normal climate were the following: beech:  $\rho=0.805 \text{ g/cm}^3$ ; black locust:  $\rho=0.810 \text{ g/cm}^3$ ; spruce:  $\rho=0.441 \text{ g/cm}^3$ ; and poplar:  $\rho=0.395 \text{ g/cm}^3$ . The change of the internal temperature takes place by the same process for each wood species. The difference is in the intensity of heating, which correlates with density. During the heating of the timber, species with lower densities warm up faster than denser species. It can also be observed that species of similar densities (poplar and spruce as well as beech and black locust) show values close to one another. Accordingly, there is a small difference between the curves for poplar and spruce, and the same can be said for the those of beech and black locust. There is however a significant difference between the two density groups.

Following the first 5 minutes of heating, the internal temperatures of all 4 wood species changed at quasi-identical paces. At 10 minutes, it can be seen that there is a more significant difference between the two groups having different densities. This difference increases for a while, then, approaching the final temperature, it continuously decreases. After 50 minutes, practically all 4 species reach the desired temperature. From this perspective, the difference is that poplar and spruce – whose densities are lower – already reached it after 30 minutes.

The influencing effect of the anatomical directions can also be well observed during heating (Figure 3). The heat flow in the longitudinal direction takes place faster than in the radial and tangential directions. There is practically no difference between the radial and tangential directions. They behave similarly both in terms of intensity and in terms of reaching the final temperature values. In the longitudinal direction, the heat flow already separates from the other two directions in the initial heating stage, reaching the desired temperature faster.

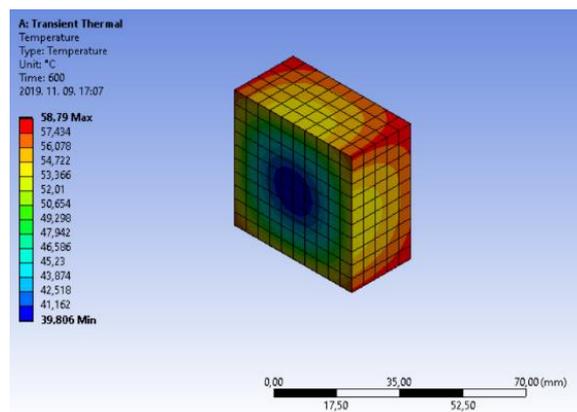


**Figure 2: Heating of the wood species tested as a function of time**



**Figure 3: The effect of anatomical directions on heating beech wood**

Modelling the process using the finite element method was greatly helped by the known actual measurement results. Figure 4 shows the temperature distribution inside the poplar timber in the 10<sup>th</sup> minute of heating. It can be seen well that heating is more intensive along the edges. While these parts reach the desired temperature at the start of heating, the temperature inside the timber is still significantly lower.



**Figure 4: Temperature status of poplar in the 10<sup>th</sup> minute of heating**

The differences between the model and the test results are shown in Table 1. The process was successfully modelled for all 4 wood species tested. It can be generally said that when modelling the duration of the heating period, differences show in the middle phase of the process, which are nevertheless not significant.

**Table 1: Difference between practical measurement and the temperature calculated by the model**

Species	Difference (°C)
poplar	1.08-3.7
spruce	1.04-3.3
beech	0.91-3.6
black locust	0.7-2.5

## CONCLUSIONS

Density and anisotropy have a significant influence on heat flow inside the timber. During the heating of the timber, species with lower densities warm up faster over their entire cross sections than denser species. As for the anatomical directions, the fastest heating takes place in the longitudinal direction, while no significant differences emerge between the radial and tangential directions. The heat flow process taking place in the timber can be modelled well using the finite element method. The differences between the measurement results and the model were between 0,7-3,7°C. The model may assist the various technological processes in the course of treating wood. The necessary technological times can thus be better determined, translating to savings in time, energy and costs.

## ACKNOWLEDGMENTS

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## Evaluation of dry heat treatment of *Platanus acerifolia* timber with special emphasis on the resistance to fungal decay

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**Keywords:** thermally modified timber, dry heat treatment, protective effectiveness, resistance to fungal decay, *Platanus acerifolia*, *Coriolus versicolor*, *Coniophora puteana*

### ABSTRACT

The aim of our research work was to compare the resistance of untreated and dry heat treated xylem of London plane (*Platanus acerifolia*) to *basidiomycetes* fungi. Plane trees are less well-known wood species in the Hungarian industry. We also aimed to try to enlarge the scientific data of London plane. The thermal modifications were carried out at 180 and 200 °C reaction temperatures. The schedules were performed according to the ThermoWood Handbook (ITWA, 2003) and the duration at the selected reaction temperature was 5 hours each time. The sapwood and heartwood boards investigated were cut out from the same log. The specimens to be treated at 180 °C and 200 °C and their control one were cut out of a board along the grain to make observable the relative change of durability. We used testing method according to EN 113. Experiments were carried out using white-rot fungus, *Coriolus versicolor* and brown-rot fungus, *Coniophora puteana*. All the schedules independent of the reaction temperature were successful in reducing the mass loss of the xylem caused by *C. puteana*. In case of *C. versicolor* only the treatments at 200 °C showed significant influence on the resistance to its decay.

### INTRODUCTION

The heat-treated wood, as a raw material discovered again is more and more used in the wood industry nowadays. The choice of the wood species was motivated by the followings. The thermally modified timber (TMT), as a raw material for the secondary wood uses no added chemicals. It's well known that the thermal modification increases the fungal decay resistance of wood and causes pleasant brownish colour, which can be enlarge the application field of the timber. The main aim of the study was to make comparison concerning the protective effectiveness against fungal decay of various treatments.



*Figure 1: Preparing (left) and conditioning of London plane timber*

## MATERIALS AND METHODS

Our investigation focused on the heat treatment of the xylem of *Platanus acerifolia*. The sapwood and heartwood specimens were cut out at the breast height of a tree harvested in the botanic garden of the University of Sopron. Three specimen groups could be defined according to the level of the selected wood modifications. Therefore the “untreated” specimens were prepared from the native xylem after conditioning at normal climate (relative air humidity = 65%, and air temperature = 20°C). Another two groups of specimens were the “treated at 180 °C”, and the “treated at 200 °C” according to the reaction temperature of the schedules. The duration at the reaction temperature was 5 hours in all cases. The dry heat treatments of the xylem were carried out in atmospheric air without addition of water or steam. The dimensions of specimens for testing the resistance to fungal decay were 50 × 25 × 15 mm according to the EN 113 standard applied (Fig. 2.).



*Figure 2: Specimens (left), and placing of specimens into Kolle-flasks (right)*

The heat-treated test specimens were sawed out from thermally modified xylem after conditioning at normal climate. According to the test groups mentioned above, three specimens from the same type of xylem (sap- or heartwood) were placed into a Kolle-flask, which were sampled from the native wood closely along the grain. The test fungi were *Coniophora puteana* and *Coriolus versicolor* with applying a standard duration of incubation (16 weeks). The measuring the mass of the specimens before and after the incubation was performed according to the EN 113 too. The rate of fungal decay can be calculated by the following formula:

$$m_{ol} = \frac{m_{obefore} - m_{oafter}}{m_{obefore}} \cdot 100 \quad (1)$$

where:

- $m_{ol}$  mass loss, the rate of fungal decay, [%]
- $m_{obefore}$  oven dry initial mass, [g]
- $m_{oafter}$  oven dry mass after the incubation, [g]

The less the rate of decay, the more durable is the wood to the enzymatic decay of the fungus.

## RESULTS AND DISCUSSION

Table 1 summarizes the average mass loss caused by fungal decay after standard incubation. The white-rot fungus, *C. versicolor* showed the highest degrading activities in case of the specimen group “untreated” with an average sapwood mass loss of 31,46 %, and with an average heartwood mass loss of 20,79 %. At the same time the brown-rot fungus, *C. puteana* caused moderated wood degradation with an average sapwood mass loss of 23,58 %, and with an average heartwood mass loss of 9,79 %. Although the schedules at 180 °C did not caused considerable change of resistance against the enzymatic attack of *C. versicolor*, the average mass loss values of both xylem types caused by *C. puteana* were very significant (<3% in both cases). These very low mass loss values could not be reached by dry heat treatments at 200 °C reaction temperature in case of *C. versicolor* (>4,5% in both cases).

**Table 1: The average mass loss of the specimens caused by fungal decay after standard incubation (EN 113)**

Mass Loss, m <sub>oi</sub> (%)	untreated	treated at 180°C	treated at 200°C
<i>C. versicolor</i> / sapwood	31.46	28.08	4.69
<i>C. versicolor</i> / heartwood	20.79	26.26	8.76
<i>C. puteana</i> / sapwood	23.58	2.48	0.56
<i>C. puteana</i> / heartwood	9.79	1.51	1.89

## CONCLUSIONS

Considering our testing results the white-rot fungus, *C. versicolor* caused the highest enzymatic wood degradation not only in native sapwood of *Platanus acerifolia*, but in case of its native hardwood too. On the other hand, the moderated mass loss of the native sapwood caused by *C. puteana* was roughly two times higher than in case of the native heartwood. It was also found, that the resistance to fungal decay of the xylem could be enhanced by dry heat treatment, but significant protective effect of the schedules at 180°C could only be observed against the enzymatic attack by *C. puteana*.

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## Wood formation genetics in poplar (*Populus*): structure and regulation of key candidate genes

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**Keywords:** *Populus*, wood property, microfibril angle, SNP markers, miRNA, regulation network

### ABSTRACT

Given their veritable industrial value, on poplars, the study of the genetic basis of wood formation has enormous potential utility. In our study, we explored the genetic background of two wood property phenotypic traits: microfibril organization and matrix deposition. Following the identification of candidate genes encoding enzymes with role in determining these aforementioned wood quality attributes, at first, on coding regions of these genes, SNP markers were developed. Our markers worked successfully across a wide range of *Populus* species/hybrids, being useful in detecting structural differences of genes related to specific characteristics. Then, a similar marker development has been performed on the promoter region of the same genes, providing molecular exploitation tools toward deciphering the cis-regulation of these genes' expression. Finally, we carried out an *in silico* analysis throughout the *Populus trichocarpa* L. genome, concerning the multigene interactions' controlled regulation of an important factor of wood stiffness, the microfibril angle. Our computational approach identified miRNAs and their target genes putatively connected with microfibril angle determination. Following gene annotation and coexpression network construction, a regulation system responsible for microfibril angle determination has been unraveled. Notably, the results indicated an interaction enrichment of auxin response target genes. Our work may come in support to wood industry not only by providing tools for the marker-assisted breeding strategy of poplars, but also by new data concerning the regulation inherent microfibril angle determination.

### INTRODUCTION

Poplars are of considerable economic value (Rademacher et al. 2017). Having significant economic interest, there is a demand for accelerated improvement strategies that aim at developing *Populus* species as wood-producing crops with both improved trunk performance and specific exploitation characteristics. Thus, for innovative selection toward a specific structure, composition and properties of the raw material, would be of great help the marker assisted breeding of the species. However, to apply genetic and molecular tools to optimize the breeding strategies, as a first step, these tools must be developed and in-depth understanding of the mechanisms that determine new or enhanced traits is required.

Until nowadays, the molecular analysis of plants often focused on several genes' structural level. Plant genomics and epigenomics are the approaches, that have reversed the paradigm of identifying genes behind biological functions, instead focusing on finding biological functions behind genes, reducing the gap between phenotype and genotype (Hartwell et al. 1999). As nowadays' biological research increasingly relies on network models to study complex phenomena, most of the attempts are in reconstructing molecular circuits that model the way how cells receive, process, and respond to information from the environment, providing snapshots of the overall cell dynamics, from the view of a particular biological phenomenon.

For wood industry, the biological event which may be of particular interest, is the formation, and as defined by the previous, the mechanics of wood. Mechanical properties of wood are influenced by a number of factors, including the anatomical structure, the walls of the constituent cells, the adhesion between them and their turgidity (Ryden et al. 2003). In their turn, mechanical characteristics of plant cell walls are largely controlled by the structure of the cytoskeleton. In terms of mechanical properties, while in the cytoskeleton

the microtubules and actin filaments are of paramount importance (Fletcher and Mullins 2010), within the cell wall, the orientation of cellulose microfibrils is the most important factor. The wood cell walls have multiple layers that differ in microfibril organization and ratios of cellulose to matrix (lignin, hemicellulose and pectin) components, microtubule-associated proteins being involved in a scaffolding between nascent microfibrils and the microtubule network (Luo et al. 2016, Sasaki et al. 2017).

It is obvious, that the whole process is complex. However, the previous attempts made to reveal the genetic background of this process by studying only a few genes' structure and neglecting regulations mediated by hormones and transcription factors, may not fully reveal the complex mechanisms of wood formation. The complexity of the whole process strongly increases, when post-transcriptional regulation is included, mediated by microRNAs interacting with different transcription factors.

Motivated by the complexity of the whole wood formation process and with aim to provide molecular tools conducting breeding strategies toward enhanced traits, conducted a two-component survey, starting from marker development on coding regions of genes with role in determining wood quality attributes (Köbölkuti et al. 2019), The second component constituted the *in silico* construction of signal transduction pathways with presence of miRNAs, transcription factors and genes, involved microfibril angle (MFA) regulation.

## EXPERIMENTAL METHODS

### ***Marker development on coding regions of genes with role in determining wood quality attributes***

#### **Search for sequence similarities and primer design**

For the development of markers putatively connected with the wood production process, a data mining was performed and proteins with possible role in the wood production process were selected based on the work of Halpin (2004), Fromm (2010), Takabe et al. (2001), Fromm and Lautner (2016), Vander Mijnsbrugge et al. (2000) and Arend et al. (2005). Based on these data, we filled a BLAST database with EST sequences downloaded from NCBI public database. The constructed EST BLAST library was searched using the BLAST Genomes toolkit (<https://blast.ncbi.nlm.nih.gov/Blast.cgi>) against the *Populus trichocarpa* (taxid 3694) database. After the search based on sequence homology, were selected the sequences, that had the highest BLAST hit by setting the thresholds lowest E-value at <0.001, greatest identity at >98% and the HSP length between 100 and 1000 bp. The selected genes' coding sequences were used for primer design. The design of primers was performed with the Primer 3 Plus software (<https://primer3plus.com/cgi-bin/dev/primer3plus.cgi>) and the primer pairs were tested *in silico* using the Primer-BLAST toolkit (<https://www.ncbi.nlm.nih.gov/tools/primer-blast/index.cgi>).

#### **Plant material**

The experiment in the laboratory was conducted with a single sample/individual, collected in the NARIC FRI ex situ gene collection plantation established in Sárvár-Bajti (Hungary). From several individuals wood tissue samples were taken by an increment borer. In some cases, samples originated from leaf material, selected from the former NARIC FRI DNA collection base. The following poplar species were involved into our investigation: *Populus nigra* L. ('Lassicsárda 7', 'Lébény 211', 'Győr 203'), *Populus deltoides* Marsh. ('Durvakérgű', 'S-1-54', 'S 307-24', 'S 332-1'), *Populus trichocarpa* Torr. et Gray ('Muhle Larsen'), *Populus alba* L. ('Villafranca'), *Populus tremula* L. ('105'), *Populus × canescens* SM. ('65'), *Populus grandidentata* Michx. ('227'). In addition to clones representing pure species or natural hybrids, *Populus × euramericana* [Dode] Guinier cultivars bred and registered in Hungary with the highest aboveground biomass yield and wood quality ('Pannónia', 'Koltay', 'Kopecky'), moreover four promising new Hungarian clones ('Sv-656', 'Sv-761', 'Sv-874', 'Sv -890'), and several clones originated from Belgium, Italy and Poland ('Beaupre', Raspalje' [*Populus × interamericana* Brockh.], 'I-214' [*Populus × euramericana*], 'Kornik-21' [*Populus maximowiczii* A.Henry × *Populus × berolinensis* Dippel]) were collected.

#### **Testing the primers in the laboratory**

For DNA extraction, one cm<sup>2</sup> of a single leaf or 10 mg core was ground to powder in liquid nitrogen. Total DNA was extracted, following a modified alkyltrimethylammonium bromide (ATMAB) protocol (Dumolin et al. 1995). As a result, 100 µl of high purity concentrated DNA solution was obtained. 50 µl of the DNA solutions were stored as a reserve at -80 °C, for possible future studies. Oligonucleotide primers were

synthesized by standard phosphoramidate chemistry at IDT (Integrated DNA Technologies (Bio-Science Ltd.) Coralville, USA).

With each primer-pair on each DNA sample a PCR test was carried out to evaluate the functionality of the designed primers. PCR was conducted by following a modified protocol of Isabel et al. (2013). The reactions contained 10 ng/μl template DNA, 1x reaction buffer (Promega GoTaq G2 Flexi, 5x Reaction Buffer with no magnesium), 2 mM MgCl<sub>2</sub> (Promega, Madison, USA), 150 μM dNTPmix (Promega, 10 mM each), 0.5 unit polymerase (Promega GoTaq G2 Flexi, 5U/ μl) and 0.13 μM of each primer (IDT) in a total volume of 15 μl. PCR was carried out in a Veriti Personal Thermocycler (Applied Biosystems, Waltham, USA), with a pre-denaturation step at 95 °C for 3 min, followed by 35 cycles of 94 °C for 30 sec, an annealing temperature for each primer combination at 50 °C for 45 sec, extension at 72 °C for 90 sec, and a final elongation at 72 °C for 10 min. The PCR conditions were considered optimal and the tested markers suitable for further sequence analysis in case of PCR-amplified sequential motifs appeared as a single band in the agarose gel. For markers with multiple bands further optimization was performed by increasing the annealing temperature up to 55 °C. PCR amplification products were analysed on a 2% agarose gel (Roti Agarose, Roth GmbH, Karlsruhe, Germany), following electrophoresis on 120 V for 1 hour, in 1x TAE electrophoresis buffer, and stained with GelRed (Biotium Inc., San Francisco, USA). From all products, those that appeared as a single band were selected by random and used to verify the previous annotation's correctness and to detect potential SNP polymorphisms.

### **Sequence analysis**

The purification of the products after PCR was done by hydrolysing the excess primers and dephosphorylated unincorporated dNTPs, in one step, with ExS-Pure enzymatic PCR cleanup kit (NimaGen BV, Nijmegen, The Netherlands), according to the manufacturer's protocol. As a next step, fragments were sequenced, the direction of sequencing being chosen considering the best BLAST hit between the reverse and forward variant of each sequence. Considering the above-mentioned direction, clone 'Pannónia' was an exception, in case of which the process was performed in both directions. Sequencing was completed at Biomi Ltd., Hungary. Visual analysis and editing of the sequences were performed with BioEdit Sequence Alignment Editor version 7.0.9.0 (Hall 1999). A second analysis was performed in case of ambiguity sequences, where the analysis of the double traces was completed by Trace Recalling method (Tenney et al. 2007), with use of the CodonCode Aligner 8.0.2 (CodonCode Corporation) software. After editing, the alignment of sequences was completed with CLC Genomic Workbench version 12.0-Viewing Mode (QIAGEN Bioinformatics, Hilden, Germany), setting gap open cost at the value of 10, gap extension cost at the value of 1, end gap cost 'as any other', and in a very accurate (slow) mode. Following sequence alignment, a final validation of all potential SNPs was made visually on chromatograms again with use of BioEdit, and sites that differentiated *Populus* clones were identified. Number of polymorphic sites, nucleotide diversity, the character of SNPs (synonymous or non-synonymous) were calculated using DNA Sequence Polymorphism version 6.10.01 (Rozas and Rozas 1995) software.

### ***In silico identification of microfibril angle (MFA) regulation***

#### **Data sources**

A list of identified candidate genes for low and high microfibril angle (MFA) has been compiled based on the work of Li et al. (2012). Based on this list, an EST library was fulfilled with publicly available ESTs (103038 sequences of eudicots), putatively related to MFA, accessed and retrieved from NCBI. The latest reference *P. trichocarpa* genome assembly (RefSeq assembly accession: GCF\_000002775.4) was downloaded from National Center for Biotechnology Information (Tuskan et al. 2006). A mature miRNA library was created with all currently available mature miRNA sequences (10414 sequences) from all plants, retrieved from MiRBase data repository (<http://www.mirbase.org/>) (Kozomara et al. 2019). A protein BLASTX database with 760,913 sequences from all plants has been generated with protein sequences accessed and retrieved from NCBI.

#### **Bioinformatics data mining**

The EST library was subjected to sequence cleaning and assembly using EGassembler (Masoudi-Nejad et al. 2006). By this method, all repetitive sequences were masked, trimmed and discarded. The vector sequences were also masked, using NCBI's vector library. Sequences having similarity to plastids and mitochondria were masked and discarded as well, using the '*Arabidopsis thaliana*' plastids and 'plants' mitochondria library. The remained sequences we assumed as high-quality ESTs and were assembled into

contigs and singletons. The search of contigs and singletons against the *P. trichocarpa* genome has been performed by the NCBI BLAST+'s BLASTN package in Linux environment, searching the contigs and singletons as nucleotide queries against the genome as nucleotide database. The best matched BLAST contig and singleton sequences were subjected against all mature miRNAs from plants, available at MiRBase data repository. MiRNAs and their potential target sites in *P. trichocarpa* were identified using a two-step strategy. At first by using miRanda, an algorithm for finding genomic targets for microRNAs (Enright et al. 2003). The second phase constituted the validation of those miRanda results, which were identified by the following BLASTX search as coding sites for proteins involved in the molecular control of microfibril angle. The validation process was accomplished by psRNATarget (Dai et al. 2018). The putative proteins involved in the molecular control of microfibril angle and which biosynthesis is regulated by miRNAs were identified and selected by mining the reference protein library of all plants using the basic local alignment search tool BLASTX algorithm with the previously miRanda detected miRNA target genes as queries. The 'blastx' application has been accomplished by Galaxy/Europe NCBI BLAST+ blastx tool. In order to understand the biological meanings of the output blastx search resulted list, GO annotation was performed using PANTHER version 14 (Mi et al. 2019). The identified targeted genes were networked using Cytoscape's GeneMANIA (Warde-Farley et al. 2010) and STRING (Snel et al. 2000) applications.

## RESULTS AND DISCUSSION

### ***Marker development on coding regions of genes with role in determining wood quality attributes***

In a first phase of our study the tested 55 primers on 23 different poplar DNA samples successfully amplified 49 sequences (success rate 89%), and 26 PCR products appeared as a single band in the electrophoresis gels (success rate 47. 27%). Our results suggest that such primers could work across a wider range of *Populus* species/hybrids. Transferability of EST markers among closely related species has been reported not only in crop species (Decroocq et al. 2003, Feng et al. 2009) but also in *Populus* (Du et al., 2013). Our values could be considered somewhat congruent with the results of similar marker transferability tests.

Fragments from the following eight genes were selected by random, for sequencing: blue copper binding protein, ptk 2, SAMS, in forward and COMT, COMT, CCoAOMT, SKOR in reverse direction. Conclusive chromatograms were obtained in case of seven gene fragments. By this method, on 23 poplar DNA samples each, fragments from seven different genes were successfully amplified and identified. After sequencing, COMT 3, ptk2/2 and SKOR 3 showed clear sequencing chromatograms only for PCR products of 21 samples each, therefore in total 164 sequences were analysed.

In the second phase of our marker development, new fragments of the genes encoding SKOR, SAMS and CCoAOMT enzymes, as well as the Araf-ase and EC3218 (endo-1,4-b-xylanase) coding sequences determining microfibril angle, were amplified, sequenced and analysed from both directions. In total, fragments of nine different genes were amplified and identified using 13 marker regions, of which seven gene segments were examined for a total of 22 different poplar genotypes. Thus, a total of 188 sequences were analysed. These results seem therefore to indicate that coding sequences functionally annotated can be amplified and utilized as genetic markers in species from the *Populus* genus, using heterologous primers. By the detection of polymorphic sites, these primers can now be more extensively tested for eventual polymorphisms that are more or less frequent in poplar hybrids both with improved trunk performance and specific exploitation characteristics.

Single nucleotide polymorphisms were found in number of 90 of 78,748 bp. The number of mutations ranged from 0 (CCoAOMT 5) to 17 (CCoAOMT 4) per sequence. Nucleotide diversity values ranged from 0.0000 (CCoAOMT 5) to 0.01109 (Kt 1), with a mean of 0.01575. Synonymous mutations were found in 25 cases, ranging from 1 to 9. The highest value was found for COMT 3 (9), with no synonymous SNPs in case of two markers. The number of non-synonymous single-base mutations (49) ranged from 0-10, with the highest value (10) for COMT 4. None of the non-synonymous SNPs generated an early stop codon. According to the literature, nucleotide diversity provides valuable insights into the genomic imprint of selection in regions of the genome with different functional characteristics (Wright and Andolfatto 2008), and into the genetic basis of wood formation, perenniality and dormancy (Brunner et al., 2004). Some of the non-synonymous SNPs detected in this study are of special interest because they might have an

influence on the protein structure and function. For example, non-synonymous SNPs found in CCoAOMT 4 could lead to the modification the composition of lignin and secondary xylem development, or non-synonymous base changes in the potassium channel SKOR encoding sequence could change the regulation of K<sup>+</sup>-dependent wood production, conformational changes in endo-1,4-b-xylanase (EC3218) may result in differences in microfibril angle. From this aspect, our markers could provide a valuable examination tool of specific alleles that contribute to poplar wood traits, a very important aspect of current Hungarian poplar breeding programs.

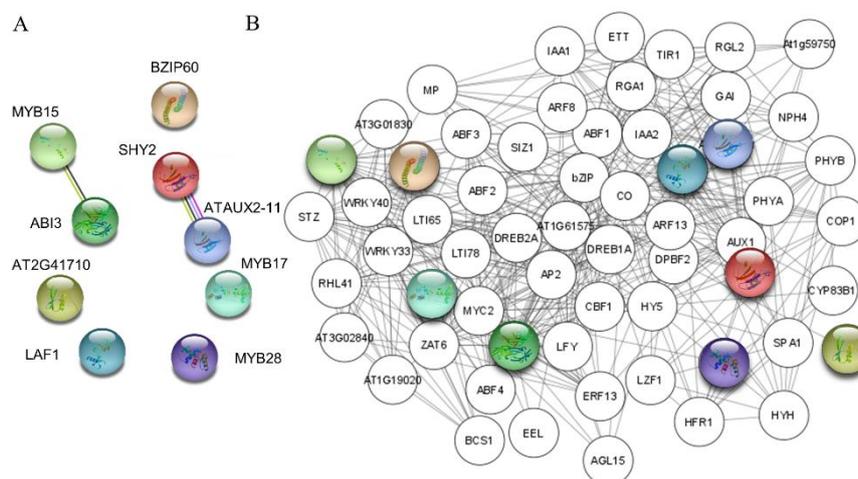
### ***In silico identification of microfibril angle (MFA) regulation***

The sequence cleaning of the EST library containing 103,172 'eudicots' sequences resulted in 103,079 ESTs, a number of 93 being trashed from our collection. All remained sequences were assembled into 12,947 contigs and 16,459 singletons. The NCBI BLAST+'s BLASTN search of all contigs and singletons in the *Populus trichocarpa* genome identified 2870 (from singletons) and 3612 (from contigs) sequences, respectively. The sequences remained as singletons we assumed to be genes expressed at low level, while the contigs with different length (between 101 and 32,75bp), genes expressed at different levels. Taking into consideration, that all our contigs and singletons derive from EST sequences in conjunction with microfibril angle determination and the reorganization of the microfibrils is tissue specific, affected by different environmental stimuli, these differences in length can be deduced from different expression levels. The *P. trichocarpa* sequences from all miRNA-target site combinations were used to mine the reference 'all plants' protein library. The BLASTX search revealed nine proteins involved in the molecular control of microfibril angle and which biosynthesis is regulated by miRNAs. From all miRanda results, by filtering and selecting the previous nine proteins' coding sequences and their regulatory miRNAs, in total 7436 miRNAs were identified as RNA molecules with role in silencing and post-transcriptional regulation of these genes' expression. By checking all these data with psRNATarget, the number of miRNAs was reduced at 397. This result leads us at two main conclusions: as first, one miRNA can exert its regulatory effect on several targets. Secondly, a specific gene can be regulated by multiple miRNA sequences. Cooperative translational control, namely, that multiple miRNAs can target the same gene, was at first predicted by Krek et al. (2005) and Lewis et al. (2005), then confirmed experimentally by Wu et al. (2010), suggesting, that neither one specific miRNA, rather the combination of miRNAs is determinant concerning the target genes' expression. Target multiplicity, read as the capacity of one miRNA to have several target genes was described also (Enright et al. 2003), and rely on the possession of the conserved miRNA binding sites (Moss and Tang 2003).

By performing a functional classification of the BLASTX resulted protein list using PANTHER, we identified 13 GO modules. The whole set of modules had as function a cellular process, which regulates transcription, dependent of DNA, in response to different stimuli, in the nucleus. Consequently, the detected proteins (and their coding sequences) are part of a process, that results in a change in state, or activity of the cell, in terms of gene expression, as a result of an organic substance stimulus. From all proteins identified and annotated, in the network of predicted associations performed by STRING, between ATAUX2-11 and SHY2 (both AUX/IAA transcriptional regulators) the presence of neighbourhood, cooccurrence, database and experimental evidence, while between ABI3 (AP2/B3-like transcriptional factor) and Myb15 (transcription factor), database and cooccurrence evidence was detected. All these proteins' biological functions depict a frame of a network, which nodes have important role in transcriptional regulation during cell differentiation, in relation with auxin-, mitochondria-nucleus and abscisic acid-activated signaling pathway, in response to abiotic (cold, water, red or far red light, sulphur, salt) and biotic (insect) factors. According to literature, auxin plays a central role in plant growth and development. Regulates cell division, cell elongation, embryo polarity, vascular differentiation, apical dominance and tropic responses to light and gravity (Woodward and Bartel 2005). However, the data from literature confirms our result concerning auxin's role in determining microfibril angle. The gravitational stimulus interaction with the IAA signal transduction pathway in aspen and Scots pine is suggested by Hellgren et al. (2004), and findings indicating, that cortical microtubules play an important role in abiotic stress-induced signaling pathways can be found in the work of Wang et al. (2011). The fact, that ethylene-dependent genes may be related to cell wall composition and microtubule orientation, is suggested by Seyfferth et al. (2019). All above mentioned literature data suggest, that our findings, namely that SHY2 and ATAUX2-11(AUX/IAA transcriptional regulators), BZIP60, AT2G41710, MYB15 (stress signal

transduction), ABI3 (response to auxin and abscisic acid), MYB17 (Activator protein 1 regulator), LAF1 (photomorphogenesis) and MYB28 (cell differentiation in response to biotic and abiotic stresses) may be important nodes in a network with role in MFA determination.

Using a wealth of genomics and proteomics data by GeneMANIA, the generation of hypotheses about our genes resulted a functional genomic dataset in which the highest FDR value was related to response to auxin. Taking into consideration this result and assuming, that our selected genes are part of a protein complex, with use of GeneMANIA a network of interactions has been constructed (Fig 1).



**Figure 1: Network of predicted associations created with [a] the detected proteins only (green line: represents neighborhood; violet line: experimentally determined; green line: text mining, light blue line: protein homology and purple blue line: coexpression evidence) and [b] additional proteins, revealed by Cytoscape's STRING application**

Surprisingly, from all nine proteins identified, only four were framed into this network (Fig. 1A). In order for each of the proteins detected to be connected (Fig. 1B), the number of additional proteins had to be increased to 60. This raises the question of why, starting from genes, that can be linked to MFA according to literature, our entire study shed light on only nine proteins partially connected. Concerning this, we would like to draw attention on two aspects. At first, our EST sequences were from eudicots, many of which are presumably not found in the *P. trichocarpa* genome. Secondly, our search was pointed out only on genes (and their coded proteins), which are subjected to negative regulation of miRNAs. From this point of view, our results may indicate the most important nodes on which a combination of miRNAs intervenes to regulate a network determining microfibril angle.

## CONCLUSIONS

We developed, tested and analysed SNP markers in order to depict candidate genes encoding enzymes with wood property phenotypic traits in different clones with importance in Hungarian poplar cultivation. Our markers along with the ongoing evaluation of the wood structural features (measurements of density, stiffness, modulus of rupture, modulus of elasticity, dimensional changes like shrinkage and swelling, fibre length and MFA) on eight clones will help to obtain a more complete characterization of the specific structure and wood composition. The network representation of intracellular biological systems, considering genes, proteins and miRNAs, with role in microfibril angle determination, are scarce in literature data. For this reason, the microfibril angle determining regulatory network has been modelled through detailed integration of molecular and biochemical properties of the regulatory factors. We also demonstrated, that auxin mediated transcriptional regulators, biotic and abiotic stress-induced factors, all subjected to negative regulation of miRNAs, have important role in microfibril angle determination.

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## The dynamic water vapour sorption behaviour of the invasive alien species in Slovenia

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### ABSTRACT

Eleven types of invasive alien species found in the City of Ljubljana were studied in the present study, to determine their moisture sorption behaviour using a Dynamic Vapour Sorption (DVS) apparatus. DVS measurements are widely used to collect water vapour sorption isotherms for wood and other cellulosic materials. They are important because water influences most physical wood properties such as mechanical properties and dimensional stability, and additionally plays an essential role in wood degradation processes. All the studied woods exhibited the Type II sigma-shaped isotherms adsorption and desorption isotherms, characterised by two bends, indicated by the low initial adsorption and substantial uptake at a higher relative humidity (above 75%) for all studied samples. This study has shown that differences in the moisture–timber behaviour exist depending on the type of wood species and that influences, to some extent, most optimal use of material.

### INTRODUCTION

Invasive alien species are a threat to protected areas, species, and habitats worldwide (Richardson et al. 2007). Indeed, invasive alien tree species may have a substantial adverse effect on native communities and ecosystem processes, and they may act as ecosystem engineers, i.e. causing changes in the physical nature (abiotic or biotic) of ecosystems (Campagnaro et al. 2018). On the other hand, these wood species offers us opportunities for interesting applications, if we understand the properties of these species. However, the study of the structure, chemical composition, and properties of invasive alien species' wood, to make better and proper use, is essential. In Slovenia and several other countries today, the removed invasive alien plants are composted or used for energetical purposes due to the lack of knowledge of wood properties and behaviour.

Hygroscopic behaviour is an inherent characteristic of wood because its cell walls contain abundant water sorption sites (hydroxyl groups) and because they can swell to accommodate the sorbed water in the cell wall (Xie et al. 2011). The sorption isotherm is of particular importance for wood and other natural materials whose moisture content and material properties experience significant changes with RH (Hill et al. 2009; Glass et al. 2018). Wood moisture content affects a host of properties such as the strength, stiffness, size (dimensional stability), thermal conductivity, heat capacity, electrical conductivity, and coefficient of friction (Glass et al. 2017) and additionally plays a vital role in wood degradation processes (Fredriksson and Thybring 2018). The relationship between the equilibrium moisture content and relative air humidity (RH) is expressed with sorption isotherms, which are obtained by progressive equilibration in the adsorption or desorption process (Lesar et al. 2009).

## EXPERIMENTAL METHODS

For this study of hygroscopic behaviour of invasive alien plants, all of the tested materials were provided from the APPLAUSE project from the urban area of the City of Ljubljana (<https://www.ljubljana.si/en/applause/>).

### *Sample preparation*

Dynamic vapour sorption (DVS) analysis was performed on eleven invasive alien species (Table 1). Samples for DVS analysis were milled on a Retsch SM 2000 cutting mill (Retsch GmbH, Germany) with a conidur perforation sieve with 1.0 mm perforations. Before the experiment, the wood chips were conditioned at  $20 \pm 0.2$  °C and  $1 \pm 1\%$  RH, until a constant mass as described in details in De Angelis et al. (2018).

### *Determination of Isotherm using Dynamic Vapour Sorption*

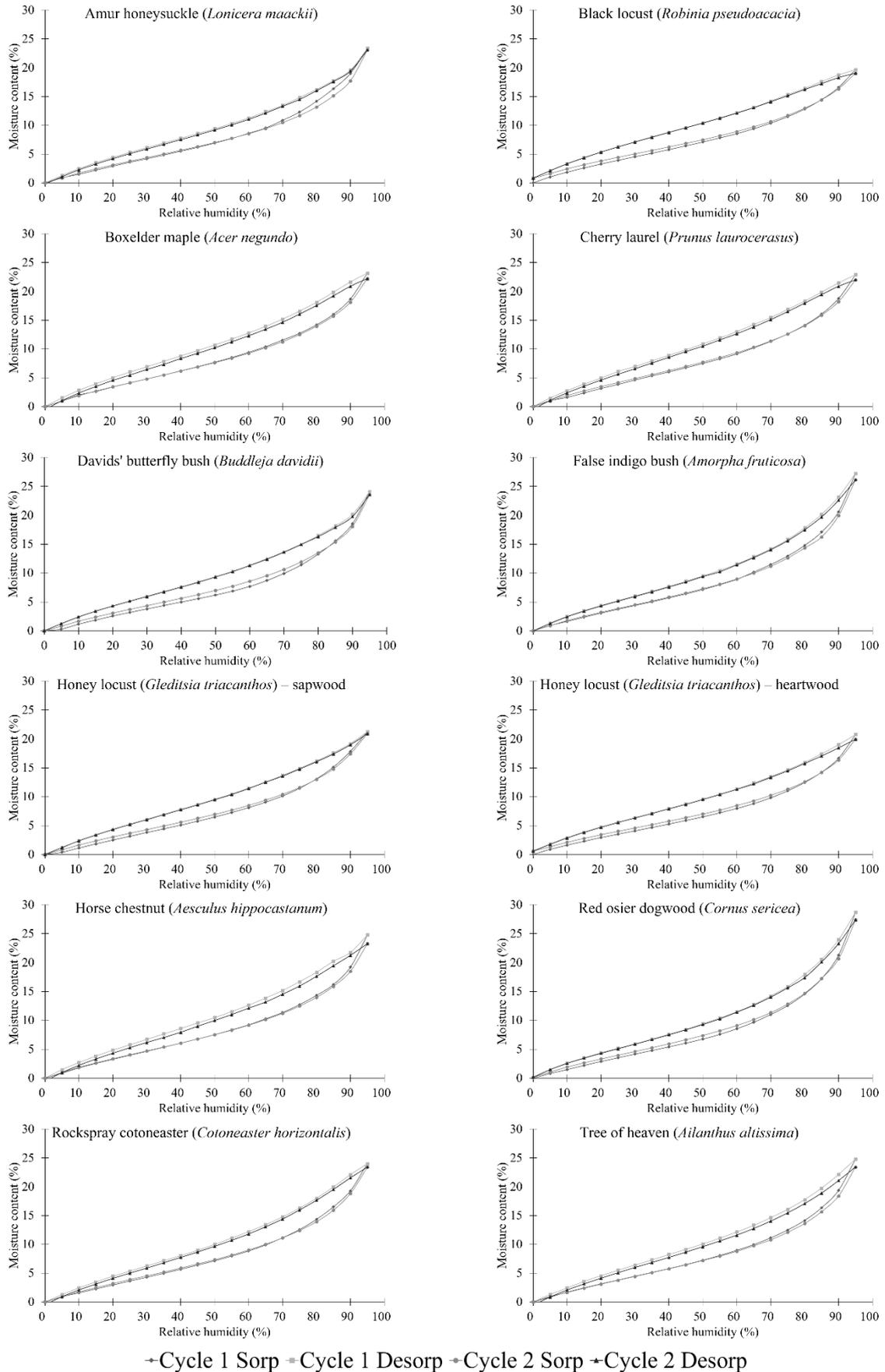
Analyses of the wood samples were performed using a DVS apparatus (DVS Intrinsic, Surface Measurement Systems Ltd., London, UK). A small amount (approximately 40-50 mg) of pre-conditioned wood chips was placed on the sample holder, which was suspended in a microbalance within a sealed thermostatically controlled chamber, where a constant flow of dry compressed air was passed over the sample at a flow rate of  $200 \text{ cm}^3 \text{ s}^{-1}$  and a constant temperature of  $25 \pm 0.1$  °C over the full RH range. The RH in the chamber was increased from 0 to 95 % in steps of 5 % in a pre-programmed sequence, before decreasing to 0 % RH in the reverse order. Two full isotherm runs were performed to capture the sorption behaviour of the material fully. The DVS maintained a given RH until the weight change of the sample was less than  $0.002\% \text{ min}^{-1}$  over a 10 min period. The running time, target RH, actual RH and sample weight were recorded every 20 s throughout the isotherm run. Sorption and desorption isotherms were produced for each material by plotting the equilibrium moisture content (EMC) change against relative humidity (RH).

## RESULTS AND DISCUSSION

Through the DVS apparatus, the relationship between moisture content against relative humidity can be determined. Two full moisture adsorption/desorption experimentally obtained isotherms for twelve samples from eleven alien woody species are shown in Figure 1. EMC at 95 % RH after first and second sorption cycle are presented in Table 1.

**Table 3: List of invasive alien woody species used in this research with equilibrium moisture content at 95 % relative humidity after first and second sorption cycle**

Species	EMC at 95 % RH (1 <sup>st</sup> cycle)	EMC at 95 % RH (2 <sup>nd</sup> cycle)
Amur honeysuckle ( <i>Lonicera maackii</i> )	23.18	23.11
Black locust ( <i>Robinia pseudoacacia</i> )	19.24	17.83
Boxelder maple ( <i>Acer negundo</i> )	22.79	22.32
Cherry laurel ( <i>Prunus laurocerasus</i> )	22.62	22.07
Davids' butterfly bush ( <i>Buddleja davidii</i> )	24.06	23.55
False indigo bush ( <i>Amorpha fruticosa</i> )	27.20	26.13
Honey locust ( <i>Gleditsia triacanthos</i> ) sapwood	21.21	20.89
Honey locust ( <i>Gleditsia triacanthos</i> ) heartwood	20.42	19.08
Horse chestnut ( <i>Aesculus hippocastanum</i> )	24.35	23.37
Red osier dogwood ( <i>Cornus sericea</i> )	28.58	27.14
Rockspray cotoneaster ( <i>Cotoneaster</i> )	23.71	23.48
Tree of heaven ( <i>Ailanthus altissima</i> )	24.47	23.53



**Figure 9: Moisture adsorption and desorption behaviour for alien woody species at 25 °C**

All the studied woods exhibited the Type II sigma-shaped isotherms adsorption and desorption isotherms, characterised by two bends, indicated by the low initial adsorption and substantial uptake at a higher relative humidity (above 75%) for all studied samples. However, there were differences in the degree of hysteresis exhibited. Highest Equilibrium Moisture Content (EMC) at 95 % RH in the first isotherm run was established for Red osier dogwood (*Cornus sericea*) at 28.58 %, followed by False indigo bush (*Amorpha fruticosa*) with 27.20 %. On the other hand, lowest EMC in the first run was measured for Black locust (*Robinia pseudoacacia*) with 23.18 %, followed with Honey locust (*Gleditsia triacanthos*) heartwood (20.42 %) and Honey locust (*Gleditsia triacanthos*) sapwood (21.21 %).

For all tested alien woody species, the EMC at 95 % RH in the second isotherm run was lower compared with the EMC at the same RH in the first run.

## CONCLUSIONS

Characterisation of sorption isotherms was achieved for eleven alien woody species using the Dynamic Vapour Sorption (DVS) apparatus. Because sorption properties can influence several characteristics of wood (e.g. mechanical properties, the performance of wood in outdoor applications, and drying properties), this paper is focused on the hygroscopic behaviour. Results on studied woods show that there are differences in the adsorption/desorption behaviour between tested alien species. Black locust (*Robinia pseudoacacia*) showed the lowest hygroscopicity in both isotherms runs, and Red osier dogwood (*Cornus sericea*) had the highest hygroscopicity among tested alien woody species. It is planned to link the sorption properties with chemical structure as well as durability in one of the future studies.

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## Durability and service life of *Eucalyptus globulus* wood in sea water in the Atlantic coast of Spain

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**Keywords:** Durability, eucalyptus, sea, marine, borers.

### ABSTRACT

In Spain, since forever damages caused by marine borers are frequent in wood elements in sea water conditions. Galicia is a region located in the North West of Spanish, above Portugal, characterized by their typical estuaries, where since ancient times, the natural production of mussels from floating wood structures. Since ever, wood species selected to build the floating wood structures mussels trough is *Eucalyptus globulus*. Currently there are more than 3,300 platforms in the sea water of Galicia. The choice of wood species with medium-high natural durability is an appropriate step of protection in the case of marine environment in sea water. *Eucalyptus globulus* heartwood is characterized by its durability against wood destroying fungi and moderately durability against marine borers, so using only heartwood it does not require preventive preservative treatment for using in a structure mussels trough, where is exposure in Use Class 3 and Use Class 5, to achieve a good performance and the expected service life. This paper shows the suitability of eucalyptus wood for its characteristics, since it combines strength and flexibility to withstand the elements and a durability of about 25 years to achieve the profitability of the installation. For its elaboration, only eucalyptus wood serves, the only one flexible and resistant enough to withstand the effects of the tides without breaking, without using preservatives and considering very well the design details and the maintenance of wood elements of floating structures mussels as key items to ensure a good durability during the estimated service life.

### INTRODUCTION

Since forever, in Spain, are frequent damages caused by marine borers in wood elements in sea water on the Spanish coast. In fact, there have been detected serious damages caused by marine borers in sea water contact wood elements located in the warm waters of the Mediterranean Sea, in the South East coast, as well as in the coldest ones in the Atlantic Ocean in the North West coast. These attacks are usually for the country's wood infrastructures and other wood elements submerged in sea water as is the case of ships, floating structures mussels trough, poles, stakes, harbours and other wood elements.

Degradations caused by marine borers depend on natural durability of wood species used and the treatment applied with water-soluble salts. In the case of softwood species as: *Pinus radiata*, *Pinus pinaster* and *Pinus sylvestris*, commonly used in coastal structures in overall Spanish coast, the durability against marine organisms is very low.

In short, that regardless of tropical wood species suitable for use in elements under Use Class 5, as for example *Ocotea rodiei*, commonly known as Green Heart or Bebeere; *Dicornya paraensis* with also named Kabakally, Angelica do Para, Teka of Guayana, etc.

Other option is using chemical modified woods, because at the moment in Spain there are no really effective chemical or physical treatments against marine borers. However, advances in Chemistry in the field of civil construction have allowed the development of protection products that, although are not specifically formulated as protectors for wood elements, seem to have good mechanical performances and durability in contact with seawater, so that perhaps they can be used as physical barriers against marine borers in the future.

So, since ever a usual solution in Spain is to use local hardwoods species in sea water conditions with a medium natural durability against marine borers as: *Quercus robur*, *Castanea sativa* and *Eucalyptus globulus*. In this sense, the paper describes the durability and service life of *Eucalyptus globulus* in sea water in the Atlantic coast of Spain, where is commonly used in floating structures mussels trough.

## DURABILITY IN SEA WATER

### *Climatic and sea water conditions*

Climatic and sea water conditions are a key aspect in the durability of wood elements in sea water, strongly affects the durability and susceptibility mainly to decays of wood and marine borers. In Spain, the levels of Scheffer Index as well as the average number of days with precipitation higher to 1mm have been defined as ones of the parameters to characterize climatic conditions.

### *Use classes*

The concept of use class is related to the probability that a wooden element is attacked by biological agents. In the case of Europe, Use classes, defined in the EN 335: 2013 are based on differences in environment exposures that can make the wood or wood-based products susceptible to biological deterioration. In Spain, CTE, Spanish Building Technical Code, indicates also the probability of the wood element is attacked by biological agents and mainly depends on moisture content.

Use class 3 are that situations which the wood elements are above ground and exposed to the weather, particularly rain and where attacks by disfiguring fungi, wood destroying fungi and wood-boring insects, including termites in the case of Spain, are possible. Decay risk depends on the climatic and other conditions as design of details, position of wood elements and maintenance provision. A large variety of situations exists and use class 3 may divided into 2 sub-classes: Use class 3.1 and use class 3.2. In the situation of use class 3.1, the wood will not remain wet for long periods, water will not accumulate. This may be achieved by, for example, by a suitable design, coating, maintenance or orientation of components to shed water or to dry quickly. In the situation of use class 3.2, the wood will remain wet for long periods, water may accumulate. This may be achieved by, for example, due a bad design, not coating or not maintenance.

It is emphasized that the approach should consider the period where a wood element remains above a 20 % moisture content, since there is no doubt that an outdoor element, without contact with the ground in use class 3 (use classes 3.1 and 3.2) in Central and South Spain (hot and dry climate conditions) have not the same risk of being moistened and consequently attacked by wood destroying fungi that the same element in a similar situation in North Spain, where wet and warm climate conditions are the most common.

Use class 5 is the situation in which the wood or wood-based product is permanently or regularly submerged in salt water, i.e. sea water or brackish water. Attack by invertebrate marine organism is the principal problem, particularly in the warm and certain salinity waters where organism such as *Limnoria* spp., *Teredo* spp. and Pholads can cause significant damages, which hollow out extensive tunnels and cavities in wood elements. Attack by wood destroying fungi and growth of surface moulds and staining fungi is also possible.

The above water portion or certain components, for example harbor piles, can be exposed to wood-boring insects.

In Europe, the occurrence of marine borers is ubiquitous. In the case of Spain, the situation is similar as in the rest of European territories, with a presence of marine borers ubiquitous in all sea water and especially in the Atlantic Ocean.

Regarding floating structures, we find depending on the wood elements different uses classes: Use class 5 in wood elements permanently or regularly submerged in salt water and use class 3 in wood elements above sea water and exposed to the weather, where even we can find wood elements in sub-class 3.1, not remain wet for long periods, and sub-class 3.2, remain wet for long periods.

### ***Durability of wood***

Wood, due to its organic nature, is supposed to be degraded and returned to nature because of the degrading action of biological and/or abiotic agents that directly or indirectly are involved in its degradation. Biological agents are composed by living organisms that degrade wood, including moulds, wood disfiguring fungi, wood destroying fungi and wood destroying insects; and abiotic agents comprising mainly atmospheric agents (sun and rain), as well as chemicals products.

Biological agents are responsible for attacks that cause reductions in the resistant of wood elements and consequently their physical and mechanical properties that will be taken into consideration when using the wood and its protection. The action of wood destroying insects is characterized by perforations and tunnels, while fungi attack produces a variety of defects, including destruction of anatomical elements.

Abiotic agents themselves do not cause serious damage. However unsuitable environmental conditions, particularly temperature and humidity in the environment surrounding the wood elements allow biological agents attacks and affect the final service life.

The natural durability of a wood species is defined as the inherent resistance to attack by wood destroying organisms, while the treatability is the ability which a liquid penetrates inside the material. The natural durability and treatability are two fundamental aspects to consider in the selection of a wood species in a wooden house.

The choice of wood species with medium-high natural durability is the first and most appropriate step of protection in the case of marine environment in sea water timber structures such as floating structures mussels trough where there are Use Class 3 and Use Class 5. However, if the natural durability is insufficient, should consider implementing a preventive preservation treatment, which increase the wood protection, in order to avoid the attack of biological agents and help keep the durability of the wood elements.

### ***Maintenance***

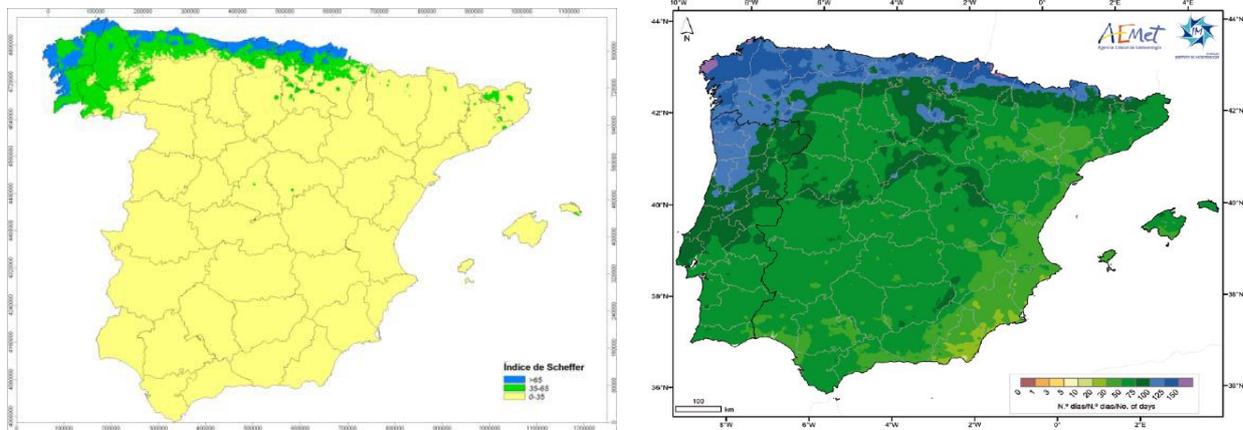
Finally, the maintenance of coating is another key item to ensure a good performance of wood exterior infrastructures exposed to weathering. An adequately coating maintenance program is necessary for assuring a correct performance.

## **DURABILITY OF *EUCALYPTUS GLOBULUS* IN SEA WATER**

### ***North West Spanish coast***

Galicia is a Spanish region located in the North West, above Portugal, with a face to the Atlantic Ocean, characterized by their typical estuaries, where since ancient times, the natural production of mussels from floating structures mussels trough in cleanness waters with an average temperature of 13°C.

Furthermore, Galicia, is, a wet and warm climate area with a Scheffer Index >65 (figure 1) and more than 125 days with precipitation higher than 1mm (figure 2). Local climate is also characterized by driven rain from south-west, high relative humidity most days of the year as well as frequent fogs.



**Figure 1 and 2: Scheffer index in Spain. Average number of days with precipitation higher to 1mm in Spain.**

### ***Eucalyptus globulus***

*Eucalyptus globulus* is a no native tree that was introduced from Australia in north Spain and Portugal in the middle of the 19th century. During the 20th century eucalyptus plantations were expanded rapidly due the pulp industry. From the first moment, eucalyptus wood was appreciated due its quality and excellent properties and used in different industrial applications as: sleepers, stakes, construction, structures, ships and floating structures mussels trough. Nowadays pulp and paper is main destination as well as another industrial uses as sawn wood, glue-lam, plywood, flooring carpentry, etc., and still is used as structural material in structures mussels trough in Galician estuaries.

In terms of natural durability, heartwood of *Eucalyptus globulus* from Galicia, Spain, is classified in the European Standard EN 350:2016 as durable (durability class 2) regarding to fungi decay and durable against wood boring beetles (durability class D), which means that its use and performance without any preservative treatment for indoor and outdoor purposes, i.e. wooden, is enough and suitable. However, although there are insufficient data available in the case of marine borers, *Eucalyptus globulus* is considered as moderately durable (durability class M) in practical experience. So using only heartwood it does not require preventive preservative treatment for using in floating structures in sea water, where is exposure in Use Class 3 and Use Class 5, to achieve a good durability and the expected service life (figures 3 and 4).

Regarding the choice of wooden materials, the wood species used not only determines the resistance to various wood destroying agents of the floating structure, also their mechanical properties, because each wood species has specific mechanical properties.



*Figures 3 and 4: Eucalyptus globulus beams attacked by marine borers.*

### **Wood structures in sea water**

Start of mussel production in Galicia can be located in the mid 18th century when the mussel came from parks or certain areas. In the 19th century, the first cultivation experiences began to occur and during the first decades of the 20th century, attempts to achieve stable cultivation in fences or stakes intensified. But it was not until the 1940s when the great development of Galician mytiliculture began, the first trough changing the cultivation of mussels on stakes for ropes suspended from a floating platform made of eucalyptus wood, where a few meters of raw esparto rope was hung. In 1970 there were more than 2,000 floating platforms throughout Galicia. In a short time, Galicia was a pioneer in the design of floating structures mussels trough, in fact, this cultivation method has become known internationally.

This innovation was helped by the availability of an important resource, eucalyptus wood, which became essential, not only due to its availability, but also due to its mechanical characteristics and durability, which had made it a wood of renowned fame among the carpenters of the riverside and because it provided long, straight and clean trunks for the construction of ever larger, therefore more productive and more durable platforms. Since ever, wood species selected to build the wood structures mussels trough is *Eucalyptus globulus*.

Currently there are more than 3,300 platforms in the sea water of Galicia (figures 5 and 6).



*Figures 5 and 6: Wood structures mussels trough in estuaries in Galicia, North West Spain.*

Generally the floating structure mussels trough is about 550 m<sup>2</sup>, approximately 27 x 22 meters, is formed by a series of floats that support a rectangular framework made up of about 20 or 25 eucalyptus wood beams on which a series of smaller eucalyptus wood pieces intersect. For the construction of a platform, between

20 and 25 eucalyptus trees are used, trees of more than 30 m are selected and are extracted from the Galician forest, to build the main and the secondary beams with which the truss of a trough is built, although some secondary beam can be obtained as long as the tree exceeds 18 m. The trees must also be very cylindrical and without branches in practically all of the first 20 m. They are looking for thick and long trunks, specimens with 40 and 50 years old, which are ideal to make the beams of the platforms, for obtaining a cross sections of all the pieces about 250 x 250 mm. without using preservatives and in some cases wood elements are finally coating with painting (figures 7 and 8).



**Figures 7 and 8: Wood structures mussels trough.**

#### ***Durability and service life of Eucalyptus globulus floating structures in sea water***

The durability and service life of a wood element is defined as the period in which it is maintained under conditions of proper use. In the case of the wood structures mussels trough in Galicia, must to be 25 years after its construction. This depends mainly on the possible damage that may appear on the parts that constitute it, decays in wood elements above sea water and attacks and degradations by marine borers as *Limnoria spp.* and *Teredo spp.* in sea water submerged wood elements.

Experts defend the suitability of eucalyptus wood for its characteristics, since it combines strength and flexibility to withstand the elements and a durability of about 25 years to achieve the profitability of the installation. For its elaboration, only eucalyptus wood serves, the only one flexible and resistant enough to withstand the effects of the tides without breaking.

Another very important item to ensure a good durability and service life of eucalyptus elements above sea water exposed to weathering (in the case of North-West of Spain, especially to rainfall) is the design details. This factor includes design details such as, exposure of the end grain, joints, etc. that avoids wet conditions may favor the attack of wood by wood destroying fungi. So the design must ensure ventilation and rapid evacuation of water in different elements, especially the main structural components and joints between beams and avoid the water traps.

The maintenance of wood elements of floating structures mussels is another key item to ensure a good performance during the estimated service life of wood exterior infrastructures exposed to weathering. A regularly cleaning of wood elements is very suitable to assure a good performance as well as a coating maintenance program is necessary for assuring a correct performance.

Currently each year about 150 punts are renewed throughout Galicia. But, despite spending 25 years floating in sea water, subjected to the enormous efforts that the sea applies to the floating structures, this story does not end there.

After serving, the eucalyptus wood used in floating platforms is still useful. Traditionally, the beams once out of the water were used as construction material. In recent years the boom in restoration of historic

buildings, or country houses, has fueled this subsequent use. Keeping its resistant capabilities almost intact and also being impregnated with sea salt that makes it difficult to attack by "terrestrial" pathogens in wood, its appearance as a large aged beam is difficult to match.

## CONCLUSIONS

From the construction, floating platforms spend during 25 years with wood elements in Use Class 3, above sea water and wood elements in Use Class 5, submerged in the sea, the durability is good, they are maintained under conditions of proper use of production of mussels, using a local hardwoods species with a medium natural durability against marine borers as *Eucalyptus globulus* wood without using preservatives and considering very well detail designs as well as maintenance during its service life.

The examples of wood structures mussels trough in Galicia with a good performance during the 25 years of their estimated service life, confirm the ability of using durable wood species against wood destroying fungi and moderately against marine borers without preservative treatments, in sea water conditions and in wet and warm climates like the estuaries of Galicia, North-West Spain, where wood destroying fungi and marine borers can develop and attack the wooden elements during the most part of the year.

Is a perfect example of possibilities about using a local hardwood species with enough durability and mechanical properties for using in sea water conditions, instead other materials as plastics, using the most ecological material, the wood.

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## The examination of seedling- and coppice black locust assortment composition in the Nyírség

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**Keywords:** black locust, assortment composition, seedling, coppice, Nyírség

### ABSTRACT

There is much debate in the forest community whether the seedling black locust or the coppice black locust is better. In our current article, we are looking for the answer to this question. We are examining and comparing the assortment composition of the seedling black locust and coppice black locust with the same origins, and how the timber management incomes of the black locust stands with different origins is changing consequently.

We did this comparison in the area of the Nyíregyházi Erdészeti, on humic sand soil and „kovárvány” brown forest soil as well. During the research we examined the clearcutting for the period 2010-16.

The primary consideration in the selection of forest areas was to have the selected stands on soils with the same production potential.

Based on the examinations, it can be stated that there is no economically significant difference between the stocks of different origins on humic sand soil (calculated at 2016 prices), however, significant differences in the assortment composition can already be observed. However, on „kovárvány” brown forest soil there are already significant differences in both assortment composition and timber management incomes.

### INTRODUCTION

Today, the black locust (*Robinia Pseudoacacia*) is the most common tree species in Hungary. Typical black locust-growing areas are mainly in the Great Plain (e.g.: Nyírség, between the Danube and the Theis rivers) and in Southern Transdanubia (Inner-Somogy).

Its rapid spread in Hungary is due to its good adaptability, its frequent and abundant seed yield, which is the basis of its excellent sprouting ability, its fast growth, and its relatively high timber yield.

The favorable physical properties and exceptional durability of black locust wood allow a wide range of applications in the area of the sawmilling industry, furniture industry and building joinery as well. It is also an important raw material for barrel production, as its wood does not allow liquid to pass through in any cutting direction. It is widely used in land engineering and hydraulics engineering, it is a durable fence post, vine pole, vine stake. Black locust is also used in the production of cellulose, fibreboard and particle board. More recently, glulam beams have also been made from it. Half of the black locust felling yield serves energy purposes, i.e. it becomes firewood.

Due to its prevalence and its widespread use it is worth to examine the assortments of stands of different origins can give, and how they affect the usability of black locust.

### TEST METHODS AND RESULTS

During our research we examined the assortment composition of seedling- and coppice black locust stands on the same site, and how, as a result, the tree utilization sales for stands of different origins develops.

We did our examinations in the area of Nyíregyháza Forestry of NYÍRERDŐ Ltd.

During our research, we examined the final harvest of 7 years, the period between 2010-16.

In the Nyírség, the black locust occurs mainly on two genetic soil types, namely humic sand soil (HH) and „kovárvány” brown forest soil (KBE).

It can be said in general about the selected subcompartments’ sites that they are found in Turkey oak-sessile oak forest climate (KTT), considering their hydrology they are independent of excess water effect, the thickness of tilth is except for one case mid-deep, and the physical soil type is sand. Most of the subcompartments belong to the middle yield class, one or two subcompartments belong to the weak yield class.

We summarized the data by village boundaries and projected per 1 ha, the resulting m<sup>3</sup>/ha data serve as a basis for comparative analysis.

In addition to the assortment composition, we also examined how the tree utilization sales of black locust stands of different origins are changing. The prices are meant to be in the woods, next to butt log and reflect the 2016 wood market situation.

## EVALUATION OF RESULTS, CONCLUSIONS

The percentage distribution of each assortment in the different village boundaries is shown in Figure 1 for seedling stands and in Figure 2 for coppice stands.

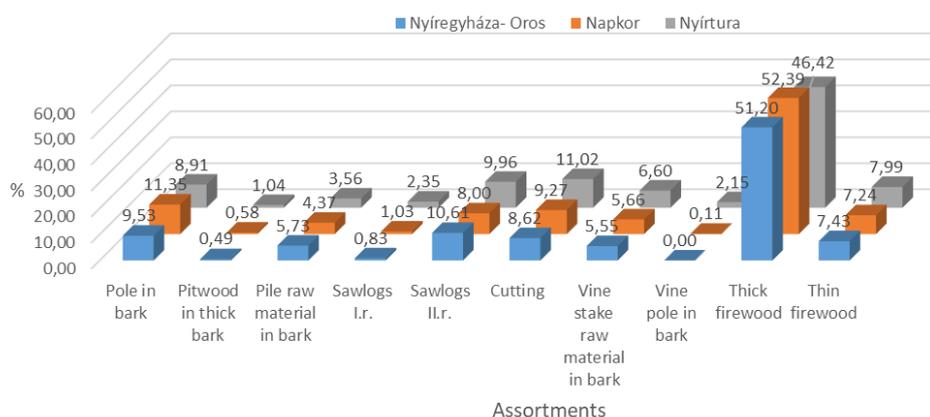


Figure 1: Seedling stands’ conversion into assortment

There is no significant difference in the assortment composition of the three border villages’ seedling subcompartments.

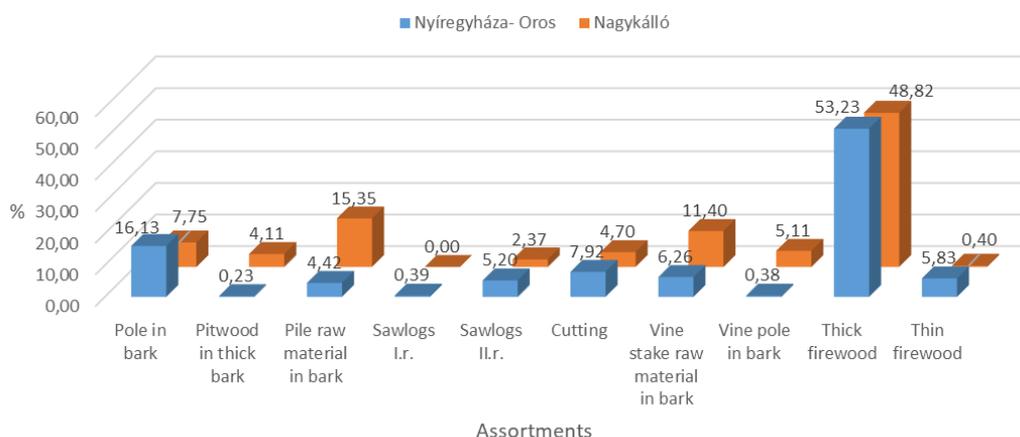


Figure 2: Coppice stands’ conversion into assortment

Figure 2 shows that the difference in the assortment composition of the coppice subcompartments of the two border villages is in the quantity of pole and pile. However, this difference is not related to site factors but to market needs, as these two assortments have such size and quality characteristics that presuppose trees with similar physiognomy.

### The first comparison

Comparison of seedling and coppice stands in case of humic sand soil site(HH). The black locust occurs in this site in the Nyírség the most often, so this comparison is maybe the most authentic. Figure 3 shows their conversion into assortments.

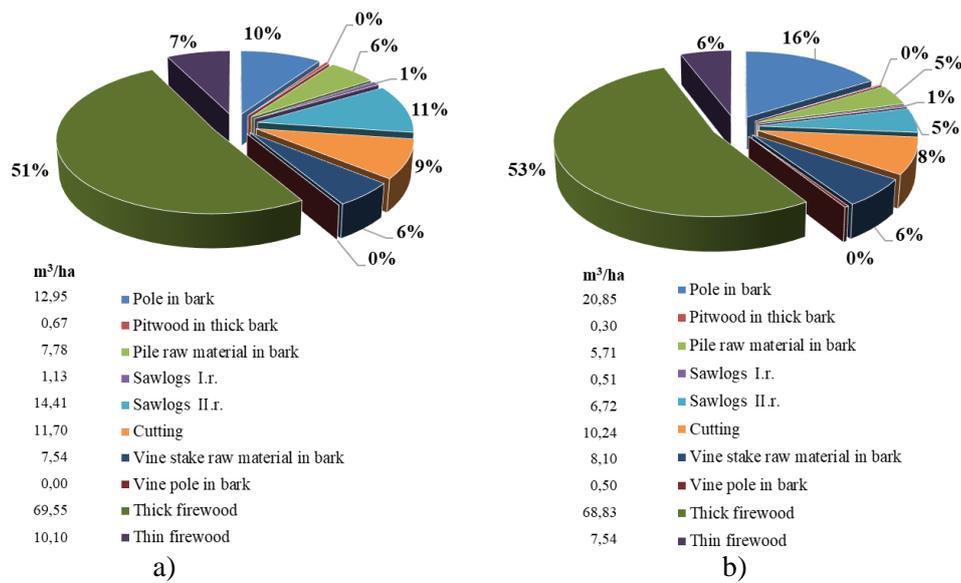


Figure 3: Distribution of the black locust stands' assortment composition on humic sand soil  
a) seedling stand; b) coppice stand

The first thing that is obvious is that in the case of the seedling stands the rate of the pole is smaller than in the case of the coppice stands. It is 10% by the previous one, which means 12,95 m<sup>3</sup>/ha, by the last one it is 16%, namely 20,85 m<sup>3</sup>/ha. The other difference shows off by the 2nd class sawn wood log. It is 11% in the case of the seedling stands, namely 14,41 m<sup>3</sup>/ha, 5% in the case of the coppice stands, namely 6,72 m<sup>3</sup>/ha. If we examine the size within the standards of these assortments, we can see that the pole can be produced up to 3-20 m long, between 14-22 cm middle diameter, whereas the 2nd class sawn wood log can be produced up to 2-6 m long, from 18 cm diameter. It makes us think that in the case of the seedling stand a bigger diameter joins to a smaller cardinal number than in the case of the coppice stand. Its reason can be found in the education of it, in the case of coppice stands the trees are getting taller because of the starting dense stand and the less living space. But in the case of the seedling stand there is a fixed interrow spacing and later it is getting thinned out, so the tree has more space to the thickness growth.

Comparing the final harvest volume of the seedling stands and coppice stands per ha, we can see that in the case of the seedling stand the wood volume is ca. 135 m<sup>3</sup>/ha, in the case of the coppice stand it is ca. 130 m<sup>3</sup>/ha.

From an economic point of view, there is also no big difference between the two origins, because the two assortments represent the same value according to the 2016 price list, so the harvest income is 2,88 millió Ft/ha in the case of the seedling stand, and 2,77 millió Ft/ha in case of the coppice stand.

### The second comparison

Also occurred between seedling stand and coppice stand, however on „kovárvány” brown forest soil (KBE). Figure 4 shows their assortment composition.

Here a definite difference can be seen in favor of the origin of the seed.

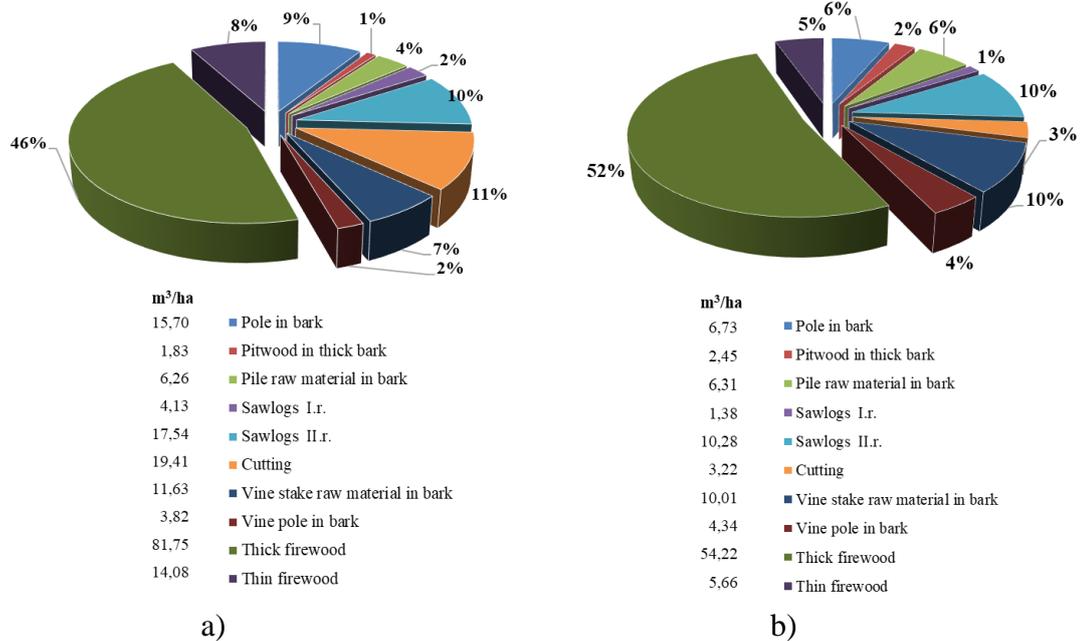


Figure 4: Distribution of black locust stands' assortment composition on „kovárvány” brown forest soil  
a) seedling stand; b) coppice stand

If we compare the final harvest volume per ha of the seedling stand and coppice stand, we can see that the difference between the two is quite big (70 m<sup>3</sup>/ha). The wood volume per ha is in the case of the seedling stand ca. 175 m<sup>3</sup>/ha, in the case of the coppice stand it is ca. 105 m<sup>3</sup>/ha.

The biggest difference is in the case of the 1st class sawn wood log (seed: 4,13 m<sup>3</sup>/ha, 2%; coppice: 1,38 m<sup>3</sup>/ha, 1%), on the other hand there is a 7 m<sup>3</sup>/ha difference in the case of the 2nd class sawn wood log in favor of the seedling stand. However, it can be seen in the case of the cutting out (seedling: 19,41 m<sup>3</sup>/ha, 11%; coppice: 3,22 m<sup>3</sup>/ha, 3%).

The tree utilization sales was 3,86 million Ft/ha, while in the case of the coppice stand it was 2,20 million Ft/ha. So there are much bigger differences in this type of soil, both in the assortment composition and in the tree utilization sales.

## ACKNOWLEDGEMENTS

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## Multi-function machine's work qualification in hardwood stands

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**Keywords:** harvester, logging damage, hardwood stands.

### ABSTRACT

Labor shortages make tree harvesting difficult nowadays. Therefore, there is a tendency that multi-function machines are spreading in Hungary. Thanks to technical and technological developments, the harvester-forwarder units can be used in broadleaf forest or thinning. Systems have part in the forest management in Hungary. However, the question is whether the quality of work will change or this system could be careful.

### INTRODUCTION

More and more harvesters and forwarders have been appearing in Hungary in the last years. Highly mechanized logging technologies are more often used, for example the CTL (Cut To Length) work system when felling, skidding, delimiting, assorting, chopping, bunching and reviewing are done with a harvester and skidding and forwarding are done with a forwarder. The timbering working systems was compared with percentage indexing methods on points developed on a conceptual or experiential basis till now in terms of the tolerance. Ormos–Rumpf–Keresztes (1990) applied a method on points, Mihály (1993) prepared weight number assessment. Suwala (2000) drew up a percentage indexing method that averages the index of the tree harms and the harm index of the upper soil layer. The damaging which can be caused by the harvesting, are not restricted to the stands of trees and the harms, which were caused in the soil. Gólya (2003) drew up an evaluative system, which takes the rest of the elements of the sylvan environment into consideration. Harvestings' complex classification can be done with the thinning result control. This method includes the examination of the tolerance.

### EXPERIMENTAL METHODS

#### *Questionnaire*

A questionnaire was carried out to find out the factors influencing the quality of harvesters working. A questionnaire was compiled in order to get to know the circumstances influencing the work, so it was possible to get the opinion of the practitioners more widely. The first few questions required independent answers (job, age, place of work), which later could be used to group the answers or to filter out irrelevant ones. The influencing factors in the questionnaire had to be rated on a scale of 1-10 (1 is the weakest 10 is the strongest). In the last reply, further comments could be made. Results in the answers are numbers from 1 to 10, so they can be easily averaged. Below the averages we calculated the standard deviation, so that the more shared questions become visible. The weighting of the influencing factors was averaged in the column direction. We averaged the answers in several directions, so we got the average value of each respondent, which is also suitable for filtering out the bad answers. Field work was aided by the influencing factors identified in the questionnaire survey. A protocol form was created. It contained possible answers to the relevant aspects, so that the conditions of the exploration under investigation should be circled only during field trips and interviews. This form allows you to categorize all the extractions according to the same criteria, and it only takes a few minutes.

**Field surveying**

The field data captures happened in Leka, Schwarzenbach and Langeck. Some of these areas were 1-5 ha private forest. Allocation of ground plots was on Borostyánkő and Kőszeg mountains. It is important to mention the 45,5 lm/ha (linear metre pro hectare) accessibility in the area of the examined forests, which is very high. To compare, in domestic forests this value ranges from 4 to 22 lm/ha, with an average of 7,2 lm/ha. The reforestation occurs naturally, the regrowth can be found everywhere, in different age distribution and density. In order to protect the natural regeneration, it is important to keep the wild game at a tolerable level, which is achieved through intensive hunting. A short-tree working system was used with harvesters and forwarders. Data measured in the plots:

- Skidding trails distances.
- Plots areas.
- Tree species, number of pieces, breast height diametres, tree heights.
- Stem hurts, hurts sizes, skidding trails deep.

**RESULTS AND DISCUSSION**

**Factors affecting loggings**

The questionnaire survey received 53 valid answers from Hungary (Figure 2) and 9 from Austria and Germany (Figure 1).

Rain	Snow	Wind	Heat	Fog	Hail	Soil	Season	Topography	Grade	Skidding trail system	Flora and fauna	Forestry/hunting	Tree species	Tree shape	Assortment composition	Stand age	Tree heights	Diametres	Density	Stem number	Task	State of mind	Personality	Experience	Motivation	Readiness for work	Working conditions	Machine type	Machine condition	Machine settings	Literature	Simulation education	Mentor	average
3	1	8	2	5	3	10	10	10	10	10			3	10	10	4	9	8	9	10		10	8	8	7	8	9	9	9	9				7,6
9	6	1	1	8	1	10	10	8	3	1	10	2	1	9	7	10	10	10	10	2	10	10	10	4	10	10	7	6	10	1	4	5	5	6,5
4	3	4	1	6	8	10	4	5	4	2	7	7	4	6	3	3	7	6	4	8	8	7	7	4	8	2	7	6	6	3	6	4	5,3	
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8	4	7	5	3	5	10	7	9	9	9	9	3	9	9	8	8	9	9	9	8	9	9	9	9	9	9	9	9	9	9	9	6	10	8,0
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2	2	3	3	2	2	2	3	2	3	3	2	3	3	1	2	2	2	3	4	3	2	2	2	2	2	3	3	3	2	3	3	2	3	1

Figure 1: The German and Austrian answers

34 manual workers and 28 intellectual workers completed the questionnaires. According to the Hungarian answers, two serious problems are clear: the low quality forestry skilled worker and the low pay of forestry enterprises. Comparing the Hungarian and German-Austrian answers, there are differences. Overall, the mean of the Hungarian answers were 7,1 and the German-Austrian answers were 6,6 so the German-Austrian respondents generally replied more modestly. As regards environmental parametres, it is generally true that Hungarian respondents considered it more important. The biggest difference can be seen in the question of ice, tin, and hail, with Hungarian respondents saying it is 37% more important. The second major difference in the issue of education is the importance of the mentor, with 24% of Hungarian respondents thinking that the mentor's help is more important. The third big difference is the characteristics of the forest. For German-Austrian respondents, wildlife is more important, 22% thought that it was more important than Hungarian respondents. There was no significant difference between the tree species, the forest equipment, tree shape, assortments. In the questionnaire, respondents provided more details besides environmental factors. According to the answers, the two most important circumstances are the person and the environment. Within a personal or machine management group, the experience, motivation and

personality influence the quality of the work more. Within the environment group, the soil, skidding trail system, topography and sloping terrain are the most influential factors, according to respondents.

Rain	Snow	Wind	Heat	Fog	Hail	Soil	Season	Topography	Grade	Skidding trail	Flora and fauna	Forestry equipment	Tree species	Tree shape	Assortment	Stand age	Trre heights	Diametres	Density	Stems number	Task	State of mind	Personality	Experience	Motivation	Readness for work	Working conditions	Machine type	Machine conditions	machine settings	Literature	Biological	Simulation	Mentor	average	
weather				environment						charateristics						personal				machine			education													
9	8	7	8	8	10	10	8	10	9	8	10	9	10	10	9	6	10	10	10	10	10	5	10	7	9	10	7	10	9	8	4	7	3	10		9
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## Circular economy principles in development of new products – APPLAUSE case study

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**Keywords:** Non-native, Woody plants, Design, Innovative products, Circular economy, Invasive Alien Plant Species

### ABSTRACT

Bioeconomy and circular economy (CE) are concept which complements each other. Wood based sector together with forest and forest-based sectors plays a central role in a bioeconomy, because it provides material as well as bioenergy. Bio-based industries have a long history of discussing and partly realizing wood cascading. On the other hand, circular economy concept and sharing economy concept are not synonyms, but they can be related. Sharing and collaborative models are one part of the picture of a circular economy. They can play a role in extending the amount of time a product is in use, while maximizing utilization.

Invasive Alien Plant Species (IAPS) are one of the biggest challenges in European ecosystems. They displace local vegetation, destroy agricultural land and cause damage to European economy in billions of euros every year. Many of them are daily removed and mainly burned. APPLAUSE project addresses unsolved questions regarding invasive alien species in terms of the zero waste approach and circular economy. By recognizing the potential of IAPS, APPLAUSE aims to introduce sustainable production processes for transforming the biomass of collected plants into useful products. Invasive alien plant species are used as a source for paper and wood products, food, production of dyes, hybrid coatings, for controlling of plant harmful organisms. Investigation of several invasive alien woody species as well as development of innovative wood products will be presented as a case study.

### INTRODUCTION

#### *Circular economy in Slovenia*

The Circular Economy is one of Slovenia's strategic development priorities. It is closely tied to the Sustainable Development Goals (SDG's) and included in key national documents such as A Vision for Slovenia in 2050 and Slovenian Development Strategy 2030 as well as in Slovenia's Smart Specialisation Strategy. The strategy's main goal is improved quality of life for everyone. Priority areas within the wider framework of natural resources: food system, forest-based value, manufacturing and mobility (Godina Košir 2018).

The transition from linear to circular models in companies can take many forms, such as:

- Circular (Eco) Design - designing products in a modular way, facilitating repairs, maintenance, modifications, restoration, dismantling, recycling, reuse, etc.
- Transitioning from products to services - the consumer becomes a user and pays for a service, while the ownership of the product remains with the manufacturer, enabling them to focus on more durable, longer-lasting products at a higher cost, with a longer life and designed according to the principles of circular design, so that the materials used can be employed as efficiently as possible, reducing the material costs and price risks.

- Industrial symbiosis - various stakeholders exchange between one another materials/raw materials that were once declared to be waste. This means that the amount of waste decreases, while the practical value of the materials increases.
- Closing energy loops - the energy surplus of one economic subject can be used as energy input by another, or perhaps it can be advantageously used within the same company. In this way, the effectiveness of energy use is greatly increased, the costs go down and the negative effects on the environment are decreased.
- Waste management systems and waste-to-energy as an important part of circular economy (Malinauskaite et al., 2017).

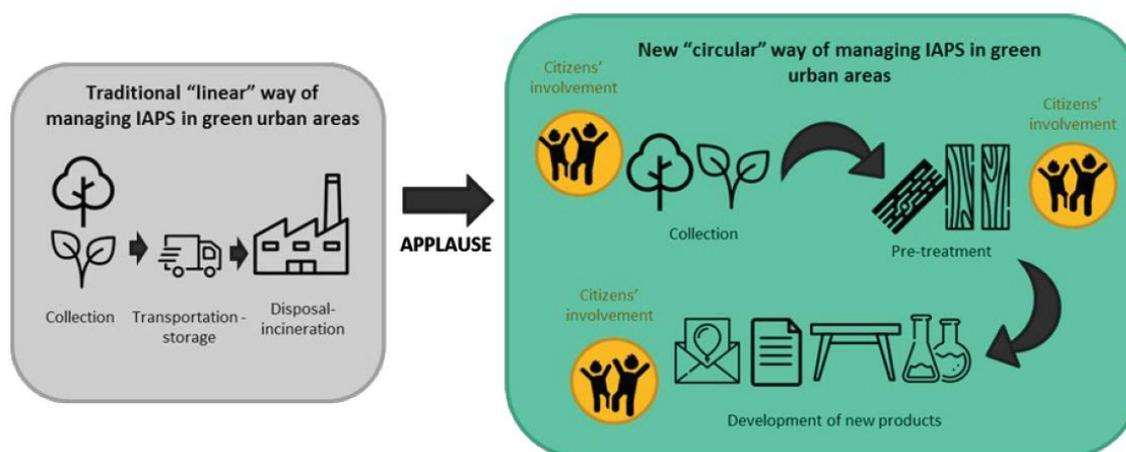
### ***Invasive Alien Plant Species and***

Invasive Alien Plant Species (IAPS) are one of the biggest challenges in European ecosystems. They displace local vegetation, destroy agricultural land and cause damage to European economy in billions of euros every year. Many of them are daily removed and mainly burned. In Slovenia there are no special landfills for invasive alien plant species, so all collected biomass is taken to incinerators. Ljubljana, as a “Zero waste city”, recognized the potential of setting up a systematic participatory model which uses collected biomass to develop new sustainable products. One of the biggest challenges is to develop successful and trustworthy circular economy models, finding new use for all parts of collected IAPS and upcycling the residual materials (MOL 2020).

## **EXPERIMENTAL METHODS**

Problematic Invasive Alien Plants (IAPS) are main topic of the APPLAUSE - Alien Plant Species from harmful to useful with citizens' led activities project. APPLAUSE addresses unsolved questions regarding invasive alien species in terms of the zero waste approach and circular economy. By recognizing the potential of IAPS, APPLAUSE aims to introduce sustainable production processes for transforming the biomass of collected plants into useful products made of wood and paper (UIA 2020).

At present plants are composted or incinerated and with the pilot project for processing plants into paper at a semi-industrial level it has been proved that this material can be utilized for other useful purposes. The proposed system of managing IAPS is based on education and cooperation with the citizens of Ljubljana and three principles of operation: »Do it yourself«; »Process with us« and »Bring to the collection centre. One of the biggest challenges is to develop successful and trustworthy circular economy model led by City of Ljubljana's leadership, finding new use for all parts of collected IAPS and upcycling the residual materials. The project addresses the widest possible circle of stakeholders: kindergartens, schools, pupils, students, households, property owners, companies, tourists, professional organizations, etc. (Fig. 1).



**Figure 1. APPLAUSE circular approach compared to the traditional circular model. (Cuixart 2018).**

## RESULTS AND DISCUSSION

### *The circular economy APPLAUSE business model*

The project introduces a completely new approach to the challenge. Invasive alien plant species (IAPS) are considered as a *resource* and starting point of a new business model. Through large-scale educational and awareness raising campaigns citizens are encouraged to participate in IAPS harvesting and use. Collected IAPS feed three main ways of their further transformation that is performed at home (e.g. food, dyes), at tutored workshops (e.g. to produce wood or paper products) and in craftsman laboratories (e.g. to manufacture innovative products with market potential in social enterprises, employing vulnerable groups). New green technologies are introduced (e.g. pilot enzymatic processing of IAPS fibers instead of chemical, reuse of wastes generated during primary wood processing and paper production, transformation of residues into liquefied wood, development of biotech-based biorafinery device for the conversion of liquor, production of 3D novel bio-composites, production of dyes, production of IAPS colored coatings, development of a model of IAPS's dye based solar cell and development of home-made formulations against plant harmful organisms) (UIA, 2020). The essence of the APPLAUSE circular business model is to:

- redefine waste materials; products need to be designed in such a way that they stay useful longer, are repairable and may be used for various purposes;
- keep raw materials in use for a longer period of time, which can be achieved by maintaining the utility value of raw materials through effective reuse, renewal and processing procedures;
- maintain and create added value of the removed biomass with a rational approach towards the use of invasive alien plants.

The circular economy business model will also be transferable to the international environment, enabling new green jobs, new skills and the inclusion of hard-to-employ people and people with disabilities (Fig. 2).

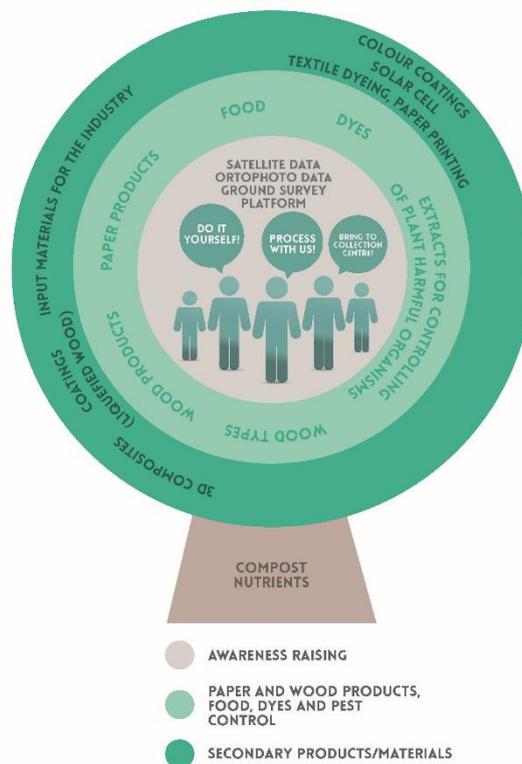


Figure 2. Circular diagram of the Applause project (by Virjent and Strojín).

The circular scheme of the APPLAUSE project (see Fig. 2) illustrates the relationship between materials, activities and products. It symbolizes a plant that is either herbaceous either wooden and represents a source of raw materials or the potential for creating benefit. Presented business model in the context of Invasive Alien Plant Species can be easily imagined in such a way that all raw materials in the process circulate and that all waste is eliminated, as this is consumed as input raw material elsewhere in the process. For example woody IAPS are used as raw material for innovative wood products such as furniture, paper products, and biochemical compounds.

**Self-sufficient business model development**

One of the goals of the European APPLAUSE project is to develop a theoretical concept of a self-sufficient business model that will be sustainable also through business and economical perspective. The conceptual business model will include preselected project activities that can contribute to self-sufficiency, will have the potential to create benefit so partners will have the capacity and willingness for further cooperation after project ends. Multidisciplinary approach is a basis to develop a self-sufficient model (Fig. 3).

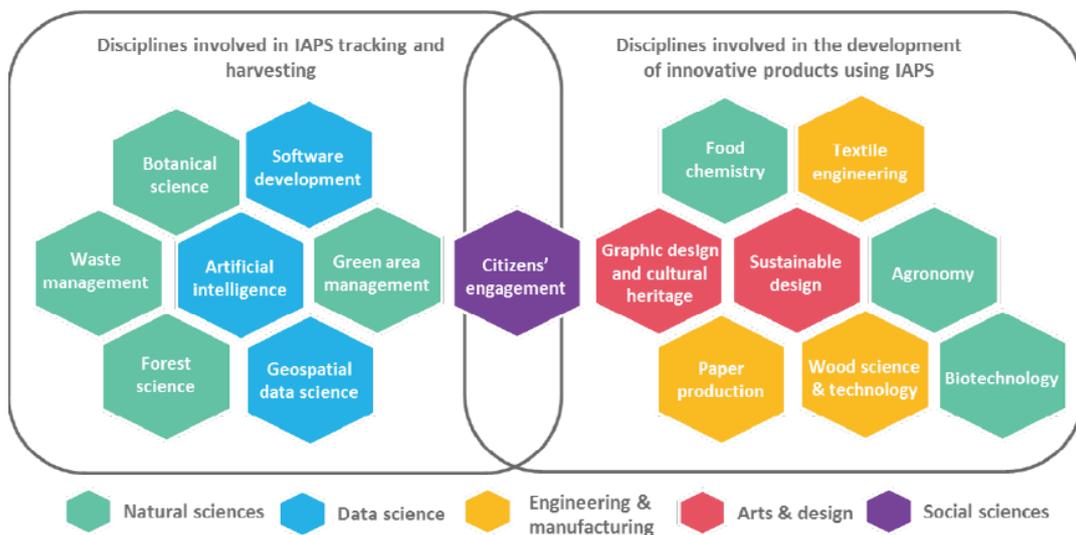


Figure 3. APPLAUSE multidisciplinary approach (Cuixart 2018).

We defined project partner’s activities and combined them in a circular business model and add partners that enabling solutions as presented in the Fig. 4.

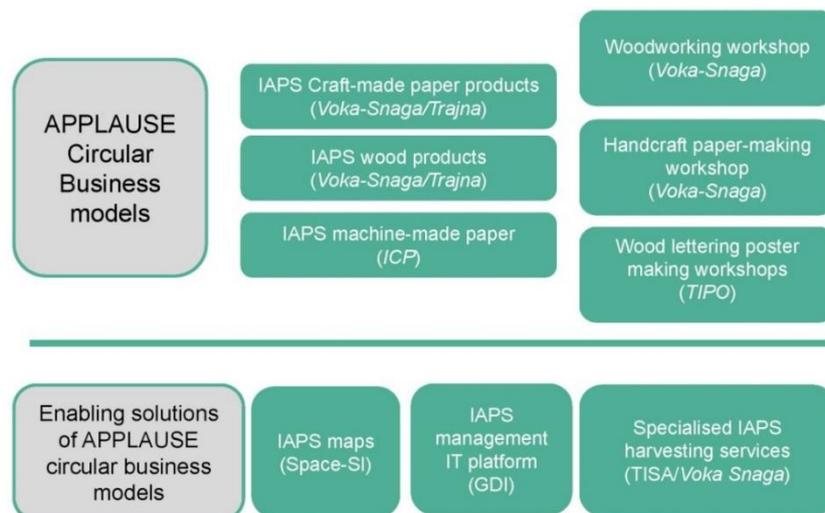


Figure 4. Proposal of Applause self-sufficient business model (by Virjent, Strojina and Cuixart).

***Future orientated innovative high value added products as a multidisciplinary result***

Innovation in design and processing of forest products is essential to meeting the challenges of 21<sup>st</sup> century forestry. Forests and attitudes toward their use are changing while demand for wood in its various forms increases with the population. Engineered wood products are among the most important, innovative wood products used worldwide (Kitek Kuzman et al. 2018) and offer new opportunities to meet consumer needs. In addition, processes such as non-destructive evaluation, recycling of wood and paper, and advances in pulping and papermaking offer new possibilities for managing the forest for its many uses (Hammett and Youngs 2002).

For innovative high added value products it is necessary to know raw material (wood) properties (Merhar et al. 2020; Merela et al. 2018, 2020; Pavlič et al. 2020; Plavčak et al. 2019). Innovative wood products have been designed as a multidisciplinary approach in practice, with activities running in parallel and partners of different fields learning to work together.

Innovative products were established as a common understanding of the project objectives and priorities; simple products were designed so that visitors to the Carpentry workshop can make them independently at home (DIY projects) or under the supervision of an experienced carpenter (Fig. 5).



***Figure 5. Innovative wood products – from construction documentation phase to prototypes.***

The purpose of innovative designed wood products is to create an aware audience that will be able to recognize the value of invasive plants and turn them into useful objects that will support sustainable life forms. As a common understanding of the project objectives and priorities sustainable products were designed as 1) simple products for users of Carpentry workshop – do it yourself at home or under the supervision of an experienced carpenter and as 2) complex products with higher added value for professional production in a specialized carpentry workshop (Fig. 5).

Detailed analyses of wood properties as well as machinability tests revealed that some woody alien invasive species are very appropriate for wood products – also products for high added value.

## ACKNOWLEDGEMENT

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## Permanent aboveground protection from termite attack on weathered envelope-treated kempas hardwood with patented cost-effective biobased pyrethroid microemulsion solutions

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**Keywords:** *Coptotermes curvignathus*, kempas, pyrethroids, termiticides, hardwood protection, envelope treatment, bio-based microemulsion

### ABSTRACT

This paper reports the excellent termite resistance in a representative hardwood species (Southeast Asian species kempas = *Koompassia malaccensis*) treated with either of the two patented biobased pyrethroid (cypermethrin, permethrin) aqueous microemulsion wood preservatives, particularly applicable to all commercial hardwoods of the world. These termiticides are approved for dipping (envelope) and pressure treatments with 25 years of termite protection for solid wood and wood-based products in Europe and more than 10 years in Indonesia. Aboveground (indoor dry conditions - H2 biological hazard class) termite field test in the Malaysian tropics determined the efficacy of such bio-based solutions, applied at three concentrations of both pyrethroids, on 3-minute envelope-treated kempas heartwood, a major hardwood material in the Malaysian wood construction market, against the Southeast Asian subterranean termite *Coptotermes curvignathus* and compared with kempas heartwood and radiata pine (*Pinus radiata*) sapwood pressure-treated with Chromated Copper Arsenate preservative. Prior to termite testing, such envelope-treated wood blocks were artificially weathered (indicative permanent use) to either (i) non-leaching/volatilization (H2 hazard class weathered wood blocks) or (ii) leaching/volatilization (H3 hazard class weathered blocks typical of aboveground outdoor wet conditions). After 6 months field exposure to termites, untreated kempas was moderately attacked (mean ratings: 7.0-7.7, mean mass loss: 17.5-19.6%), while none of the leached-volatilized (H3 hazard class) or non-leached-volatilized (H2 hazard class) test blocks treated with those bio-based products at both target retentions were regarded as attacked (mean ratings: 9.8-10, mean mass loss: <1%) regardless of pyrethroid retention, H2- or H3-weathered treated wood. Due to their unique compositions, the bio-based microemulsion termiticide solutions presented here conferred excellent, cost-effective, permanent protection against termite attack even at low concentrations and retentions of cypermethrin and permethrin compared with that for traditionally formulated solutions required for envelope-treated hardwood protection of aboveground wooden structures against Southeast Asian *Coptotermes* subterranean termites.

### INTRODUCTION

Much interest is now focused on protection against termites of solid wood or wood composite dip-treated, brushed-on or sprayed-on (collectively termed envelope treatments) with traditional emulsifiable concentrates, suspension concentrates or light organic solvent formulations often using pyrethroid and other organic termiticides at high dosages and/or without prior artificial weathering of freshly treated wood (Sornnuwat et al 1994, Peters and Creffield 2003, Donath et al 2008, Sukartana et al 2009, Tawi and Wong 2016). Termite testings of these termiticides were also evaluated as pressure-treated wood when increased termiticide penetration into the wood was desired (Creffield et al. 2013, Scown and Creffield 2009). A new generation of bio-based microemulsion termiticidal (pyrethroid-based) formulation technology with KO@LIB antioxidant additive (Messaoudi et al. 2018) has now emerged from the laboratory of Adkalis

(Berkem Group, France), popular in Europe for envelope (dip)- and pressure-treatment of wood and wood-based products. These solutions have been recently considered in Indonesia using the lowest emission costs (eco-costs) Life Cycle Assessment (LCA) methodology (Siswanti Zuraida et al. 2016). Also such enveloped-treated wood even conferred up to 8 mm cypermethrin penetration into the wood (Ruel et al. 2015). With expertise and knowledge in biocidal formulations, especially for wood preservation for over 50 years, Adkalis patented the first microemulsion technology for dipping treatments in Europe. Water-borne products can, with practical dipping or aspersion treatments and adequate dipping times and effective concentrations, also enhance durability performance of tropical hardwoods against termites under Malaysian H2-H3 biological hazard class conditions (Wong 2004) found in the humid and sub-tropics and also under warm temperate climates where severe termite threats exist. A summary of the key results of field trials conducted in Malaysia to evaluate the performance of this microemulsion technology of bio-based (pyrethroid) solutions on envelope-treated Malaysian hardwood kempas against *Coptotermes curvignathus* is reported here.

## EXPERIMENTAL METHODS

The field trials were undertaken by The Wood Biodeterioration and Protection Laboratory, University Malaysia Sarawak, Sarawak/Malaysia, using an established H2-hazard class aboveground termite field test protocol (Wong 2005, Xuan et al. 2017), meant to accelerate termite infestation (and exclude fungal mold growth and decay) on a sheltered (shielded from sunlight and water) and 1-m high stack of termite-infested keruing (*Dipterocarpus* sp.) wood at a well-drained suburban open-spaced field site where subterranean termites *Coptotermes curvignathus* are prevalent. *Coptotermes curvignathus* is representative of the aggressive subterranean termites found attacking construction wood in Malaysia and much of Southeast Asia.

**Table 1: Bio-based microemulsion solutions (over 60% bio-based content according to E.U norm NF 16640, 2017 and International norm ASTM D 6866, 2018) - Properties and concentrations used**

Microemulsion termiticide product	Initial concentration of termiticide in product [%w/w]	Diluted product concentration [%]	Diluted termiticide concentration [%]
SARPECO® AXIL® XILIX®	Cypermethrin (CMT): 1.6%	5	CMT: 0.08
		10	CMT: 0.16
		20	CMT: 0.32
	Permethrin (PMT): 1.0%	5	PMT: 0.05
		10	PMT: 0.10
		20	PMT: 0.20

The Malaysian hardwood selected for the envelope preservative treatment and termite testing was the termite-susceptible kempas (*Koompassia malaccensis*) heartwood (Wong 2000) of wide commercial structural use in Malaysia and Indonesia. Replicated air dried test blocks [2 x 2 x 5 (long.) cm] of kempas were dipped for three minutes, into one of three diluted product concentrations (or concentrations of termiticides cypermethrin or permethrin) of cypermethrin product (Adkalis 2018a) and permethrin product (Adkalis 2018b) respectively as listed in Table 1. Freshly dip-treated blocks were air dried for 2 weeks. To simulate long-term use of treated wood aboveground indoors/outdoors, treated and untreated blocks were next subjected to artificial weathering to predisposed wood blocks to the Malaysian H2-H3 biological hazard class conditions (Wong 2004) before termite testing: a 10 day full vacuum (volatilization) treatment at 40°C (blocks weathered aboveground indoor exposure = H2 hazard class) or EN84 (EN 1997) leaching regime followed by volatilization (blocks weathered aboveground outdoor exposure = H3 hazard class). Untreated control blocks and referenced volatilized and/or leached blocks pressure-treated to about 5.6 kg/m<sup>3</sup> Chromated Copper Arsenate (CCA) retention [radiata pine (*Pinus radiata*) sapwood, kempas heartwood] meant for Malaysian H2 hazard class were also included for comparison. These untreated and treated and 2 types of weathered wood blocks were exposed to the H2 hazard class field test of (Wong 2005) for up to 6 months to target subterranean termites, *Coptotermes curvignathus*.

After six-month field exposure to termites, test blocks were retrieved, adhering mixed termite carton matter-bait wood debris-bait cardboard debris carefully removed, and the cleaned blocks were visually rated for degree of termite attack on the 10-point AWP A E7-07 scale (Table 2), and oven dried (105°C) percent mass loss of wood blocks also determined (AWPA 2008). Data were interrogated by ANOVA using MINITAB-14 software, with multiple comparison t-tests of mean values (for termite rating, percent mass loss) by Least Significant Difference (LSD, P<0.05) to examine the influence of leaching, volatilization and combinations of weathering effects, and termiticide concentrations/retention on enveloped hardwood protection against termites.

**Table 2: AWP A E7-07 termite rating scheme (AWPA 2008)**

Rating	Description
10	Sound
9.5	Trace, surface nibbles permitted
9	Slight attack, ≤3% of cross sectional area affected
8	Moderate attack, 3-10% cross sectional area affected
7	Moderately severe attack and penetration, 10-30% of cross sectional area affected
6	Severe attack, 30-50% of cross sectional area affected
4	Very severe attack, 50-75% of cross sectional area affected
0	Failure (destroyed)

## RESULTS AND DISCUSSION

The 3-minute dipping (envelope treatment) of air dry kempas heartwood with 3 prescribed nominal concentrations of each termiticide yielded low mean retentions of each pyrethroid adsorbed on to the wood surfaces (calculated as either g/m<sup>3</sup> or %w/w) shown in Table 3. Notably, the lowest pyrethroid concentrations applied here (0.08% cypermethrin, 0.05% permethrin) were considerably less than that normally applied (eg. Read and Berry 1984, Sornnuwat et al. 1994, Tawi and Wong unpublished data 2019), yet conferred envelope wood protection (Figs. 1 - 8) due to the unique properties of both these microemulsion formulations of Adkalis compared with traditional formulations.

**Table 3: Mean termiticide retention (g/m<sup>3</sup>, %m/m) of envelope treated kempas based on uptake calculations**

Initial concentration of termiticide product [%w/w]	Diluted termiticide concentration [%w/w]	Mean termiticide retention	
		[g/m <sup>3</sup> ]	[%w/w]
Cypermethrin: 1.6% w/w	0.08	18.07 (12.37)	0.0022 (0.0014)
	0.16	40.54 (32.19)	0.0047 (0.0038)
	0.32	67.28 (14.14)	0.0077 (0.0015)
Permethrin: 1.0% w/w	0.05	8.06 (1.24)	0.0009 (0.0002)
	0.10	17.50 (3.26)	0.0019 (0.0004)
	0.20	36.21 (4.39)	0.0039 (0.0005)

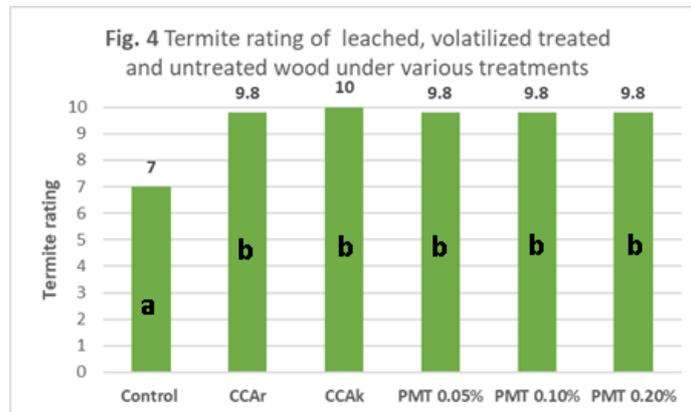
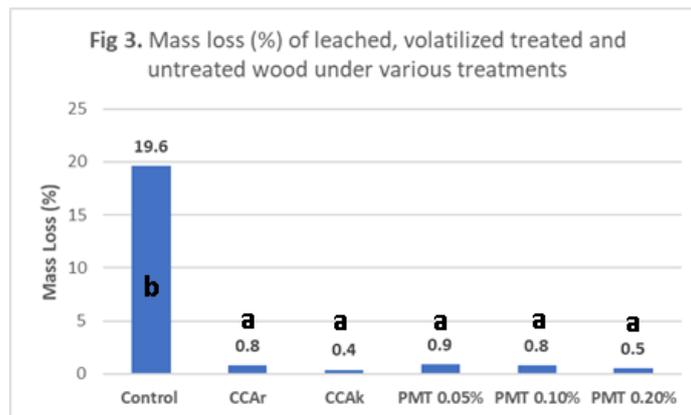
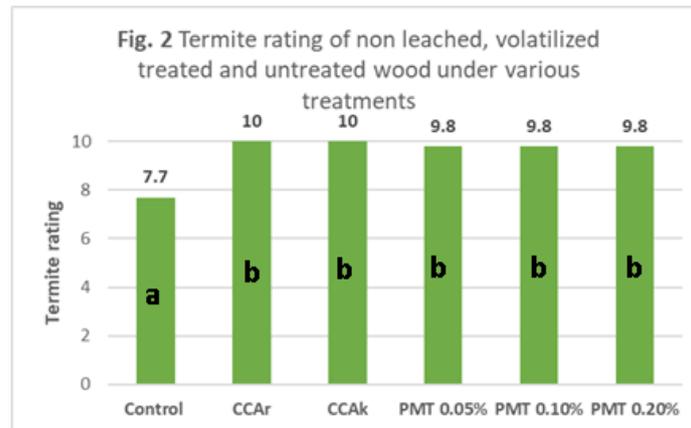
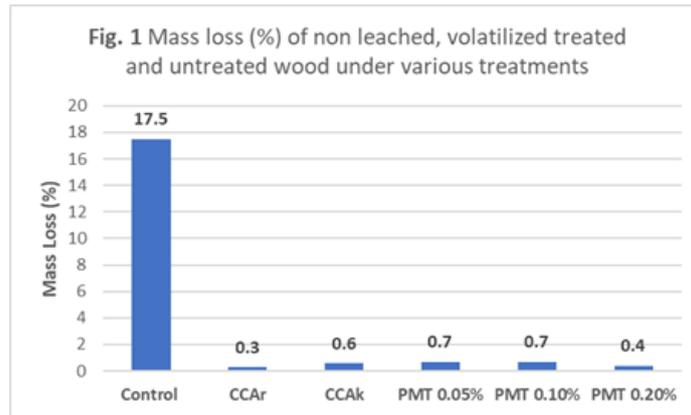
n=24; (...) = standard deviation

Applying traditional formulatons, Read and Berry (1984) revealed that a 0.1% concentration of cypermethrin emulsion using surface application was sufficient against *Reticulitermes* termites. Zaidon et al. (2008) found that exposure of rubberwood particleboard, empty fruit bunch (EFB) particleboard and Rubberwood-EFB particleboard sprayed with 0.2% permethrin solution yielded low mean mass loss (range: 7.2 – 12.1%) though failed to confer complete protection, unlike their untreated susceptible counterparts (range: 17.8 – 31.1%) against *Coptotermes curvignathus*. Excellent protection was reported from a laboratory evaluation of 5-min dip-treated rubberwood blocks exposed to *Coptotermes gestroi* at 0.015, 0.25 and 0.5% cypermethrin and at 0.5, 1 and 2% permethrin (Sornnuwat et al. 1994). Recognizing that such laboratory screening tests are not necessarily comparable with field termite tests which are realistic to

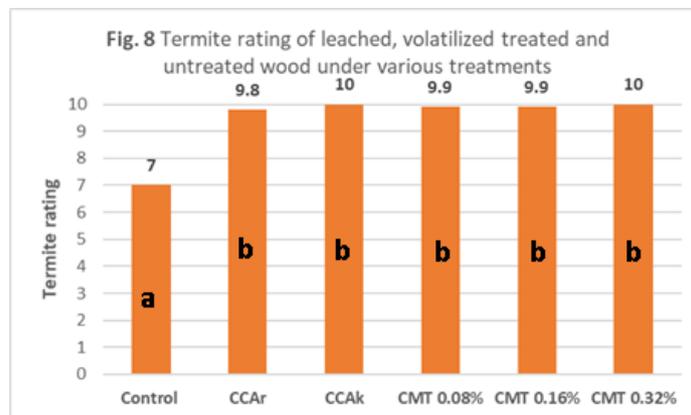
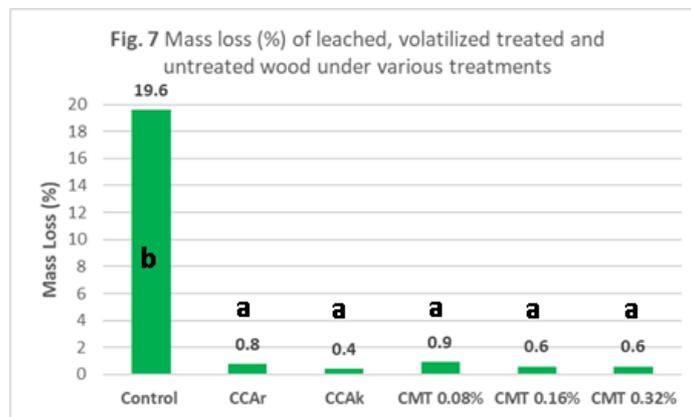
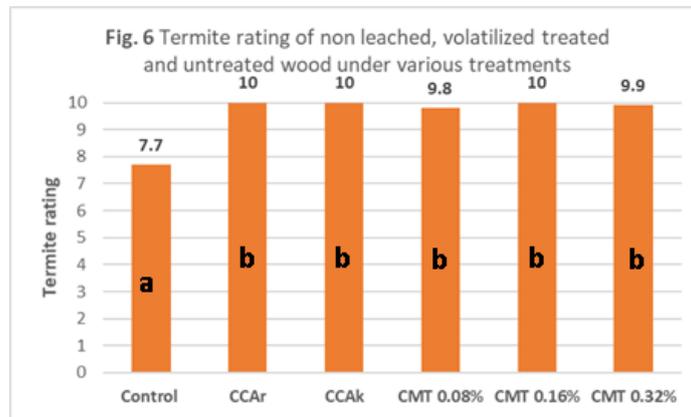
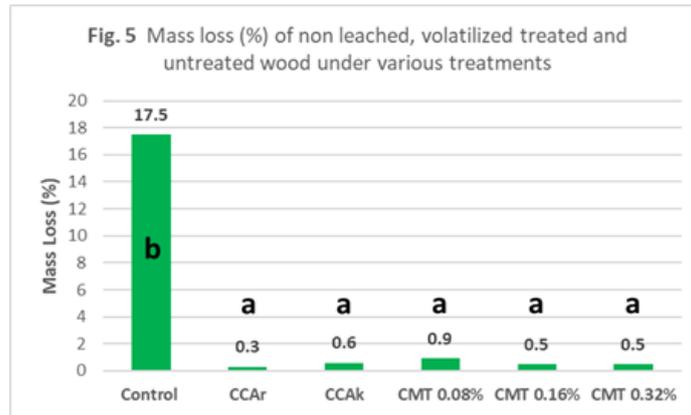
wood protection applications, H2-hazard class termite tests (Tawi and Wong unpublished data 2019) revealed instead a relatively higher levels of both emulsifiable concentrate-based permethrin (1.69 - 6.75%) and cypermethrin (1.68 - 3.35%) agropesticides needed to fully protect hardwoods from *C.curvignathus* attack. In the present field trial, 3 minutes dipping time recommended by Adkalis, protected weathered treated kempas remarkably well against termites under H2 and H3 hazard classes even at their lowest pyrethroid concentrations of such microemulsion formulation solution. However under similar conditions, traditional pyrethroid formulations (eg. emulsifiable or suspension concentrates) could confer termite resistance of dip-treated wood either at longer dipping (steeping) durations, and/or increasing pyrethroid concentrations with consequent termiticide retentions in wood (Sornnuwat et al. 1994, Kamdem et al. 1996, Ma et al. 2013). Since preservative performance against wood-degrading organisms obviously depends mainly on wood species, target preservative retention (derived from concentrations and treatment methods used) and penetration of the preservative into the wood, the unique envelope treatment microemulsion technology can provide up to 8 mm pyrethroid penetration into the wood (Ruel et al. 2015) for termite durability performance of wood aboveground contact when adequate dipping times and insecticidal concentrations are applied.

The observed surface pyrethroid %w/w retentions (determined by solution uptake trials) on kempas (Table 3) freshly treated with all 3 concentrations for each pyrethroid were extremely low (cypermethrin: 0.0022, 0.0047 and 0.0077% w/w; permethrin: 0.0009, 0.0019 and 0.0039% w/w), yet protected H2- and H3-weathered kempas well (Figs. 1 - 8) against termites. It should be ascertained if such low retentions of a pyrethroid extended to 8 mm below kempas wood surface as reported for Scots pine treated with cypermethrin in such microemulsion formulation (Ruel et al. 2015). Contrasting, the Australian Standard (2005) for preservative treatment of sawn and round timber specifies a relatively higher minimum retention of cypermethrin (0.03% w/w) and permethrin (0.02% w/w) within the 5 - 8 mm penetration zone especially for LOSP-based double-vacuum treated wood for H2-and H3-hazard class uses. Hence the present termite test appear to yield overall a promising eco-friendly and cost-effective innovative termiticide product against both the traditional water-borne and LOSP-based pyrethroids.

Figs. 1 and 2 show respectively, mean percent mass losses and mean termite ratings of untreated, permethrin-treated and CCA-treated H2-weathered woods. Figs. 3 and 4 show respectively, mean percent mass losses and mean termite ratings of untreated, permethrin-treated and CCA-treated H3-weathered woods. Both weathering regimes have not affected termite resistance of kempas envelope-treated with permethrin-based microemulsion formulation probably ascribed to the microemulsion formulation providing an effective mode of pyrethroid fixation in the wood up to 8 mm depth. Permethrin-treated (regardless of permethrin concentration) and CCA-treated woods were similarly resistant ( $P < 0.05$ ), with untreated kempas significantly less resistant than treated woods (Figs. 1 - 4). Such permethrin-treated woods, as well as CCA-treated kempas and radiata pine, were highly termite resistant compared with untreated kempas shown for H2-weathered (negligible percent mass losses and 9.8 - 10 (=highly resistant) termite rating for permethrin-treated and CCA-treated *cf* 17.5% mass loss and 7.7 (=moderately resistant) termite rating for untreated) and H3-weathered (also negligible percent mass losses and 9.8 - 10 (=highly termite resistant) termite rating for permethrin-treated and CCA-treated *cf* 19.6% mass loss and 7 (=moderately resistant) termite rating for untreated) materials. The same excellent anti-termitic performance of cypermethrin-treated woods are shown for H2-weathered (Figs. 5 - 6) and H3-weathered (Figs. 7 - 8) woods probably ascribed to strong fixation of cypermethrin in the wood sub-surface, as revealed for such treated Scots pine (Ruel et al. 2015). Overall, these results (Figs. 1- 8) revealed that the applied pyrethroid concentrations were effective and comparable to those generally confirmed efficient against *Reticulitermes* (Adkalis 2018a, b). Notably weathered envelope-treated woods displayed remarkably superb protection against termites, comparable to CCA preservative, even at their lowest pyrethroid concentrations applied, due to their unique (patented) bio-based formulations with strong pyrethroid fixation ability.



Figures 1-4: Permethrin-based, CCA treated and untreated wood compared. Mean values with same letters are not significant ( $P < 0.05$ ) by LSD



Figures 5–8: Cypermethrin-based, CCA treated and untreated wood compared. Mean values with same letters are not significant ( $P < 0.05$ ) by LSD

## CONCLUSIONS

These termite test results confirm the excellent performance using dip (=envelope) treatments of the two pyrethroid-based microemulsion formulations to confer permanent (H2- and H3-hazard class) aboveground protection of the tropical kempas wood from termites. The bio-based water-based solutions, presented in this paper, have unique and original compositions (undisclosed) that ensure effective treatment of all commercial permeable and refractory hardwoods of the world, and can be particularly suited for large-scale envelope treatment of plantation-grown hardwoods cost-effectively. Furthermore, for complete H3-hazard class wood protection which requires prevention of decay by Basidiomycetes, these formulations have been already supplemented with compatible biocides.

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## Optimization of pressing mode of particleboards with beech raw material in the composition

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**Keywords:** particleboards, beech raw material, optimization.

### ABSTRACT

In order to produce particleboards (PBs) of highest quality, coniferous tree species and soft hardwood tree species are preferred although, pursuant to standards, all tree species are suitable for their production. Very often, beech particles are also included in the composition of the boards, especially in countries from South and Central Europe where this tree species is wide-spread.

In this paper, the pressing mode in production of PBs produced from a mixture of coniferous and beech particles to the ratio of 50:50 has been optimized. UFR (urea-formaldehyde resin) with concentration of 65% and PFR (phenol-formaldehyde resin) with concentration of 32% have been used in the role of a binding agent, and as a hardener – 20% solution of ammonium chloride has been used. Under laboratory conditions, 14 main experiments and one – 15-th additional experiment have been performed according to a matrix of a planned experiment B3 at three varied factors with levels in encoded form “-1”, “0” and “+1” at following levels:

- temperature of press platens ( $X_1 \equiv t$ , °C) respectively 160 °C; 180 °C; 200 °C;
- pressing pressure ( $X_2 \equiv P$ , MPa) respectively 1.8 MPa; 2.2 MPa; 2.6 MPa;
- pressing duration ( $X_3 \equiv \tau$ , min) respectively 7 min; 10 min; 13 min).

The following properties of the produced laboratory boards have been tested pursuant to valid standards: bending strength; internal bond; water absorption after 2 and 24 hours; swelling after 2 and 24 hours. The results obtained have been subjected to variation-statistical processing and an accuracy factor below 5% has been obtained. By means of the computer programme QstatLab, the coefficients of the regression equations for the individual properties of the boards have been determined. The analysis of the results of the investigations has been performed on the single-factor cross-sections of the response surfaces of the regression equations derived. On the basis of the results obtained, the pressing mode has been optimized with respect to the indicators investigated, conclusions have been drawn, and the respective recommendations have been made.

### INTRODUCTION

Particleboards (PBs) find wide application in furniture industry and construction mainly because of the fact that they have some advantages to solid wood, viz.: possibility for use of waste wood raw material for their production; high dimensional stability; identical physical and mechanical properties in all directions of the board plane; possibility for production of boards with previously set properties; low price, etc. Pursuant to the standards, all tree species are suitable for use to produce PBs, but according to a number of investigations, most suitable for this production are the coniferous and soft hardwood tree species. Very often, however, particles of hard hardwood tree species, e.g. beech, especially in countries from South and Central Europe where this tree species is wide-spread, are also included in the composition of the boards. The goal of the submitted research work is to optimize the pressing mode in production of PBs produced from a mixture of coniferous and beech particles.

## EXPERIMENTAL METHOD

Under laboratory conditions, PBs from a mixture of coniferous and beech particles to the ratio of 50:50 and with dimensions 400x400x12 mm have been produced. A mixture of UFR with a concentration of 65% and PFR with a concentration of 32% at a ratio of 50:50 has been used in the role of a binder, and for a hardener a 20% solution of ammonium chloride has been used. The experiments have been conducted according to a matrix of a planned experiment B3 with 14 main experiments and a 15<sup>th</sup> additional one at three varying factors with levels in encoded form “-1”, “0” and “+1” at following levels:

- temperature of press platens ( $X_1 \equiv t$ , °C) respectively 160 °C; 180 °C; 200 °C;
- pressing pressure ( $X_2 \equiv P$ , MPa) respectively 1.8 MPa; 2.2 MPa; 2.6 MPa;
- pressing duration ( $X_3 \equiv \tau$ , min) respectively 7 min; 10 min; 13 min).

Following properties of the produced laboratory boards have been tested pursuant to the valid European norms and methodologies: bending strength; internal bond strength; water absorption ; swelling after 2 hours of immersion in water. The results obtained have been subjected to variation-statistical processing and an accuracy factor below 5% has been obtained.

Results for the effect of the investigated factors on the physical and mechanical properties of the boards have been processed after the methods of the regression analysis, with an experimental-statistical model of the following type having been deduced:

$$Y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_{12}X_1X_2 + B_{13}X_1X_3 + B_{23}X_2X_3 + B_{11}X_1^2 + B_{22}X_2^2 + B_{33}X_3^2 \quad (1)$$

where  $\hat{Y}$  is the output value predicted by the equation;

$B_0, B_1, B_2, B_3, B_{12}, B_{13}, B_{23}, B_{11}, B_{22}, B_{33}$  – regression coefficients;

$X_1$  – encoded values of temperature of press platens;

$X_2$  – encoded values of pressing pressure;

$X_3$  – encoded pressing duration.

Results have been processed with a specialized software QstatLab 6.0. Optimization has been performed in order to achieve maximum good physical and mechanical indicators of the boards.

## RESULTS AND DISCUSSION

Obtained arithmetic mean values of the indicators investigated are presented in Table 1, and in Table 2 – the values of regression coefficients for the target parameters. The statistical analysis made has shown that  $F_0 \leq F$ , i.e. the models deduced are adequate and may be used for prognostication of the values of the respective indicators.

Results obtained from the optimization performed are presented in Fig. 1, Fig. 2 and Fig. 3, from which it becomes clear that:

Maximum value for bending strength (15 N/mm<sup>2</sup>) is obtained at hot pressing temperature of 191.15 °C, pressing pressure of 2.60 MPa and pressing duration of 12.44 min (12 min and 26 s).

Maximum value for internal bond strength of 0.564 N/mm<sup>2</sup> is obtained at hot pressing temperature of 195.72 °C, pressing pressure of 2.12 MPa and pressing duration of 12.95 min (12 min and 57 s).

Maximum value for water absorption after immersion in water for 2 hours – 80.68%, is obtained at hot pressing temperature of 199.83 °C, pressing pressure of 2.17 MPa and pressing duration of 12.93 min (12 min and 56 s).

Maximum value for swelling in thickness after immersion in water for 2 hours – 25.18%, is obtained at hot pressing temperature of 199.34 °C, pressing pressure of 2.60 MPa and pressing duration of 11.11 min (11 min and 7 s).

Minimum value for water absorption after immersion in water for 24 hours – 94.15%, is obtained at hot pressing temperature of 199 °C, pressing pressure of 2.39 MPa and pressing duration of 12.91 min (12 min and 55 s).

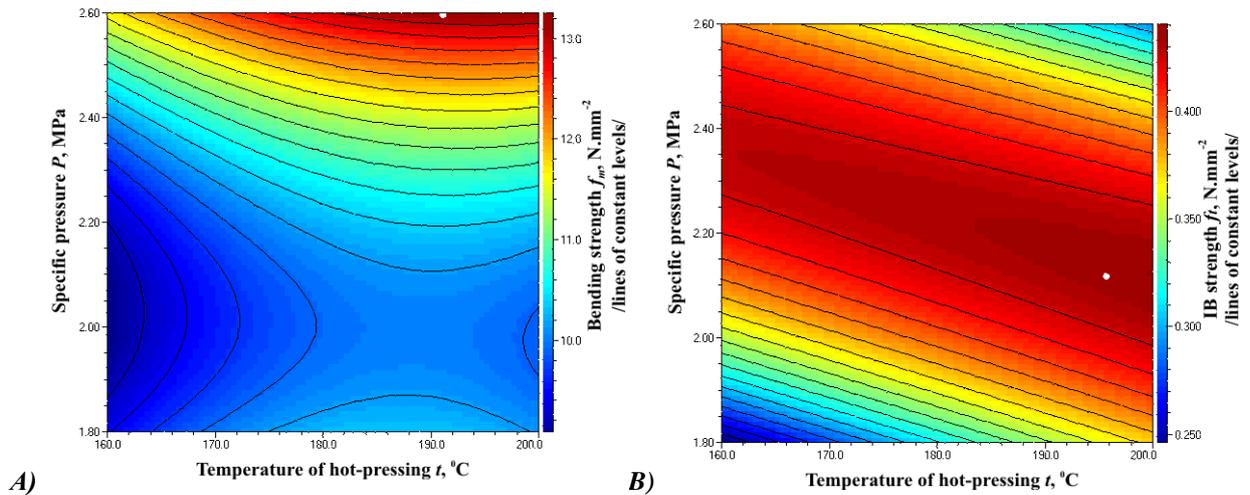
Minimum value for swelling in thickness after immersion in water for 24 hours – 25.55%, is obtained at hot pressing temperature of 198.86 °C, pressing pressure of 2.51 MPa and pressing duration of 12.60 min (12 min and 36 s).

**Table 1: Arithmetic mean values of the indicators investigated**

No.	$Y_1$ [N/mm <sup>2</sup> ]	$Y_2$ [N/mm <sup>2</sup> ]	$Y_3$ [%]	$Y_4$ [%]	$Y_5$ [%]	$Y_6$ [%]
1	8.220	0.238	151.020	60.586	147.960	75.150
2	8.932	0.312	121.160	45.650	134.897	47.830
3	10.083	0.498	105.213	35.403	117.230	39.130
4	11.500	0.316	110.594	46.610	127.133	49.870
5	10.660	0.352	118.750	38.733	128.800	40.450
6	11.208	0.549	96.730	29.250	115.380	25.400
7	13.864	0.403	92.066	25.637	104.440	23.133
8	15.291	0.395	83.548	26.064	89.437	28.330
9	10.597	0.417	90.360	30.829	105.216	31.767
10	9.274	0.409	99.762	30.672	108.870	38.673
11	12.400	0.339	98.310	29.253	114.907	32.567
12	11.080	0.311	118.445	35.443	129.213	42.767
13	11.220	0.505	93.400	31.254	110.963	32.330
14	9.740	0.432	108.967	42.296	124.530	49.507
15	10.066	0.496	94.237	37.293	118.367	47.603

**Table 2: Values of regression coefficients**

Coefficient	Values of coefficients of regression equations in coded form for:					
	$Y_1 \equiv f_m$ [N/mm <sup>2</sup> ]	$Y_2 \equiv \sigma_{ib}$ [N/mm <sup>2</sup> ]	$Y_3 \equiv A^{2h}$ [%]	$Y_4 \equiv \alpha_{\delta}^{2h}$ [%]	$Y_5 \equiv A^{24h}$ [%]	$Y_6 \equiv \alpha_{\delta}^{24h}$ [%]
$B_0$	10.167	0.434	98.807	33.256	116.073	40.905
$B_1$	0.519	0.006	-6.591	-1.850	-4.091	-3.723
$B_2$	1.304	0.019	-11.637	-4.670	-10.310	-5.857
$B_3$	1.379	0.041	-11.246	-7.961	-10.273	-11.184
$B_{11}$	-0.776	-0.012	1.702	0.354	-4.364	-0.287
$B_{22}$	1.548	-0.093	8.427	0.101	6.561	-1.563
$B_{33}$	0.288	0.050	1.233	4.528	2.247	1.689
$B_{12}$	0.198	-0.058	6.092	4.507	2.673	7.289
$B_{23}$	0.357	0.046	2.063	0.993	1.476	2.449
$B_{13}$	0.019	0.037	-0.757	-0.666	-3.158	0.841
$F$	8.397	9.475	12.642	8.287	10.411	8.434
$F_0$	4.772	4.772	4.772	4.772	4.772	4.772



**Figure 1: Variation of bending strength and IB (internal bond) strength**  
 A) Bending strength; B) internal bond strength

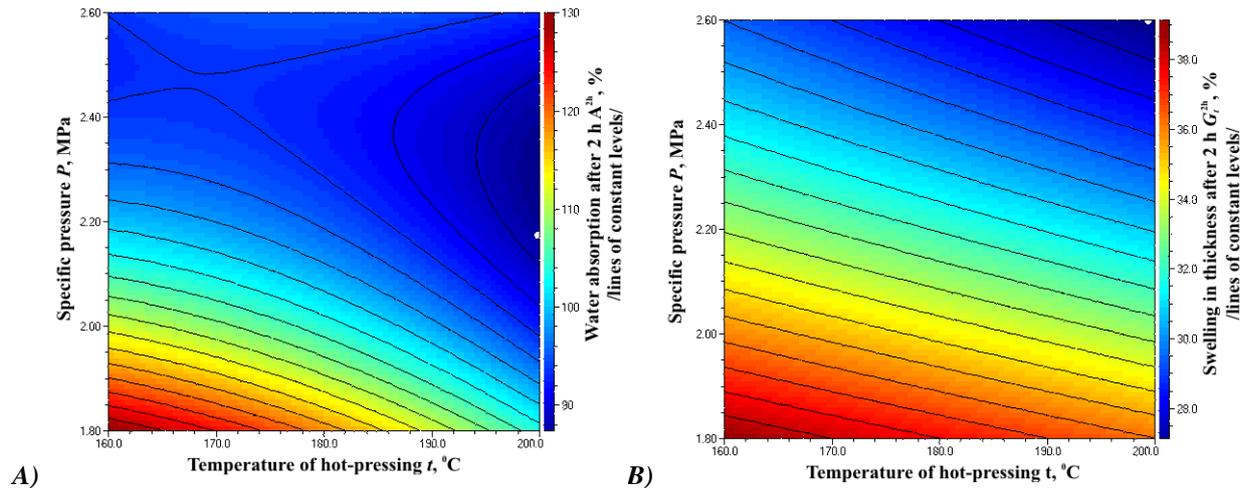


Figure 2: Variation of water absorption and swelling in thickness after immersion in water for 2 h  
A) Water absorption; B) Swelling in thickness

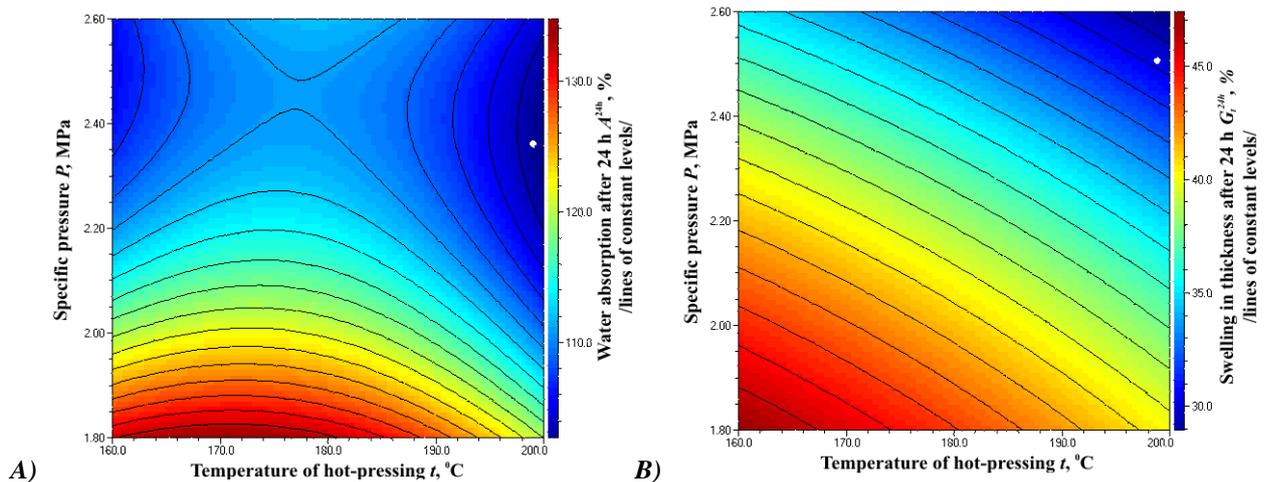


Figure 3: Variation of water absorption and swelling in thickness after immersion in water for 24 h  
A) Water absorption; B) Swelling in thickness

## CONCLUSIONS

On the basis of the investigation performed and having in mind that leading are the mechanical properties in the selection of PBs for use, the following pressing mode may be recommended as optimum one: hot pressing temperature of 191.15 °C, pressing pressure of 2.60 MPa and pressing duration of 12.44 min (12 min and 26 s).

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## Effect of content and concentration of urea-formaldehyde resin in production of medium density fibreboards (MDF) from wood of hard hardwood tree species

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**Keywords:** MDF, hard hardwood wood raw material, urea-formaldehyde resin

### ABSTRACT

In production of MDF from hard hardwood tree species, some difficulties resulting mainly from the low compaction factor during production of boards arise. These difficulties can, at least partially, be overcome with control of the factors during MDF production. Urea-formaldehyde resins are mainly used in this type of production. This is mainly due to their price and to their relatively quick polymerization under the conditions of hot pressing. When determining the resin content, its effect on the performance of the boards and the production cost price shall be taken into consideration. Another essential factor in MDF production is the concentration of the adhesive solution. This concentration shall be such that it retains the binding properties of the resins and allows their uniform distribution in the composition of the wood-fibre mass, taking into account the water content of the wood-fibre mat.

In this paper, an investigation on the effect of content and concentration of urea-formaldehyde resin on some indicators of thin MDF from wood of hard hardwood tree species has been presented. The boards are with set density of 750 kg/m<sup>3</sup>. The urea-formaldehyde resin content varies from 6% to 14% at variation of the adhesive solution concentration from 40% to 50%. The effect of these factors on the investigated indicators of the boards has been analyzed. Regression, experimental-statistical models for the effect of the amount and concentration of binding agent have been derived. The values of these factors have been determined in order to achieve maximum good values of the investigated indicators of the boards. Optimization has been performed in order to achieve the indicators of the boards, required pursuant to the standard, at possibly minimum urea-formaldehyde resin content. On this basis, conclusions have been drawn and recommendations have been deduced.

### INTRODUCTION

MDF production is one of the most rapidly growing productions in recent years (FAO). In this kind of production, wood of coniferous and soft hardwood tree species is mainly used as wood raw material (Thomanen et al. 2010). There also successful experiments, although at laboratory stage, to produce MDF entirely from wood of hard hardwood tree species (Roffel E, et al. 2013), of course, with modification of the defibration modes (Shnider, T. Et al. 2019) or of the technological modes for production of boards (Mihajlova, J. and Savov, V. 2016). Difficulties in production of MDF from this type of wood raw material exist, with them being mainly caused by the decreased compaction factor (Thomanen et al. 2010). Nevertheless, the raw material from hard hardwood tree species may be successfully used in production of HDF (high-density fibreboards) (Mihajlova, J. and Savov, V. 2018 a; b). Increased use of small-sized wood from hard hardwood tree species, which is now mainly used for firewood, will contribute to additional development of the industry for production of wood-based panels (Neykov, N et al. 2013; Neykov, N. et al. 2018) by means of extension of their raw material basis and reduction of their expenses for raw material (Neykov, N et al. 2014). In Bulgaria, and on a worldwide scale, there is a considerable raw material potential of small-sized wood from hard hardwood tree species (Savov, V 2007; Trichkov, N et al. 2005; Roffel et al. 2013), which may be successfully utilized in this production.

UFR (urea-formaldehyde resin) is mainly used in the role of a binder in MDF production (Gribsby, W.J. and Thumm, A. 2011; Gul W., et al. 2019). This is due to its relatively low price and the accelerated polymerization under the conditions of hot pressing (Kumar, R. N. and Pizzi, A. 2019). Of great importance for the MDF quality are the UFR content and its concentration of introduction into the wood-fibre mass (Donaldson, L. and Lomax T.D. 1989).

In this paper, an investigation on the effect of UFR content and concentration of adhesive solution in production of MDF from wood of hard hardwood tree species has been presented.

**Materials and methods**

To produce MDF, factory-made (after the Asplund-method) wood-fibre mass provided by Welde – Bulgaria AD has been used. The mass is composed of wood of common beech (*Fagus sylvatica* L.) – 70%, and Turkey oak (*Quercus cerris* L.) – 30%. Mass moisture content is 11.23% at bulk density of 28 kg/m<sup>3</sup>. Urea-formaldehyde resin manufactured by Kastamonu Bulgaria AD has been used as a binder. To determine the effect of urea-formaldehyde resin content, it has been adopted that it varies at three levels – 6%, 10%, 14%. The adhesive solution concentration has been changed at two levels – 40% and 50%. The paraffin content is 1% relative to oven-dry wood-fibre mass. The boards are with set density of 750 kg/m<sup>3</sup> and thickness of 6 mm. The hot pressing factor is 60 s/mm at hot pressing temperature of 190 °C. Pressing has been realized at three stages – 1st stage: specific pressure of 3.0 MPa and duration of 20% of the whole cycle; 2nd stage: specific pressure of 1.2 MPa and duration of 30% of the whole cycle; 3rd stage: specific pressure of 0.85 MPa and duration of 50% of the whole cycle. For hot pressing, a laboratory press of type PMS ST 100, Italy, has been used. The investigated indicators of the boards have been determined according to the requirements of the respective European norms (EN 310, EN 317, EN 323).

Results for the effect of the investigated factors on the physical and mechanical indicators of the boards have been processed after the methods of the regression analysis, with an experimental-statistical model of the following type having been deduced:

$$\hat{Y} = B_0 + B_1 \cdot X_1 + B_2 \cdot X_2 + B_{12} \cdot X_1 \cdot X_2, \tag{1}$$

where  $\hat{Y}$  is the output value predicted by the equation;

B<sub>0</sub>, B<sub>1</sub>, B<sub>2</sub>, B<sub>12</sub> – regression coefficients;

X<sub>1</sub> – encoded values of resin content;

X<sub>2</sub> – encoded values of concentration.

Results have been processed with a specialized software QstatLab 6.0. Optimization has been performed in order to achieve maximum good physical and mechanical indicators of MDF, with step-by-step optimization with application of 1000 iterations having been used.

## RESULTS AND ANALYSES

Results for the investigated physical and mechanical indicators of MDF at various urea-formaldehyde resin content and various adhesive solution concentration are presented in Table 1.

**Table 1: Physical and mechanical properties of MDF at various content of urea-formaldehyde resin and concentration of the adhesive solution**

Board No.	Content of urea-formaldehyde resin <i>P</i> , %	Concentration of the adhesive solution <i>C</i> , %	Density $\rho$ , kg/m <sup>3</sup>	Water absorption <i>A</i> , %	Swelling in thickness <i>Gt</i> , %	Bending strength <i>fm</i> , N/mm <sup>2</sup>
1	6	40	747±21	89.37±5.46	36.83±2.07	22.15±1.35
2	10	40	765±27	78.24±3.94	26,30±1.29	29.40±1.68
3	14	40	751±14	68.80±4.80	18,87±1.14	39.03±2.71
4	6	50	752±17	111.15±10.49	46.76±2.87	16.72±1.11
5	10	50	761±31	90.85±5.93	28.41±1.28	24.79±1.65
6	14	50	759±32	69.03±2.59	23.08±1.05	28.90±1.83

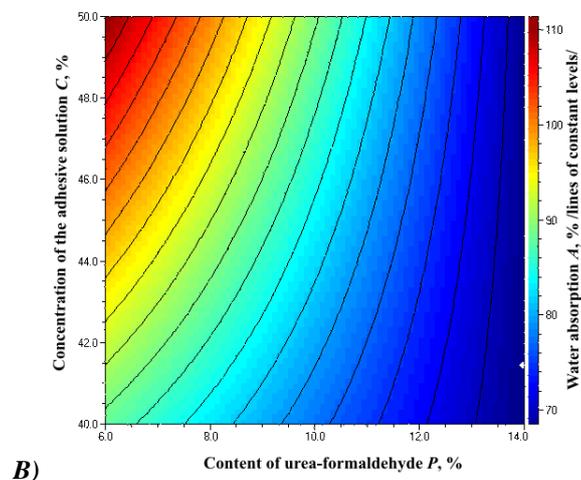
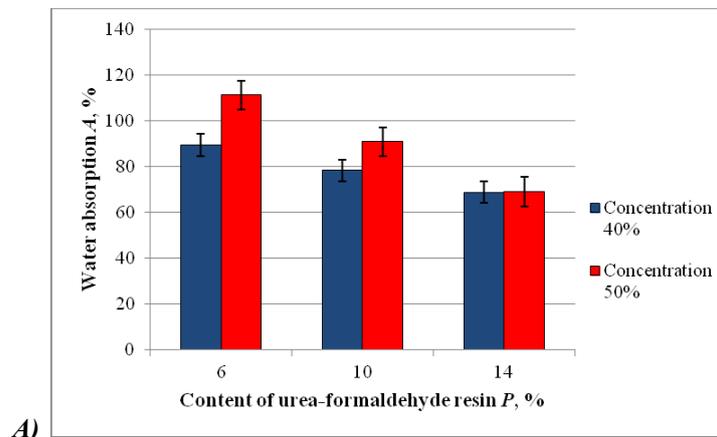
The maximum deviation of the densities of the produced boards from the set density is 15 kg/m<sup>3</sup> or deviation of 2% that is considerably below the statistical error. Therefore, the difference in this main indicator of the individual boards should not affect the remaining physical and mechanical indicators of MDF.

Regression models for the effect of UFR content and adhesive solution concentration on the physical and mechanical indicators of MDF are presented in Table 2.

**Table 2: Data from regression analysis for the effect of the content of urea-formaldehyde resin and concentration of the adhesive solution on selected properties of MDF**

MDF property  Indicator of the regression model	Water absorption	Swelling in thickness	Bending strength
Free term $B_0$	84.57	30.04	26.83
Coefficient of linear term $B_1$	-15.67	-10.41	7.27
Coefficient of linear term $B_2$	5.77	2.71	-3.37
Coefficient of interaction $B_{12}$	-5.39	-1.43	-1.18
Coefficient of determination $R^2$	0.99	0.94	0.98

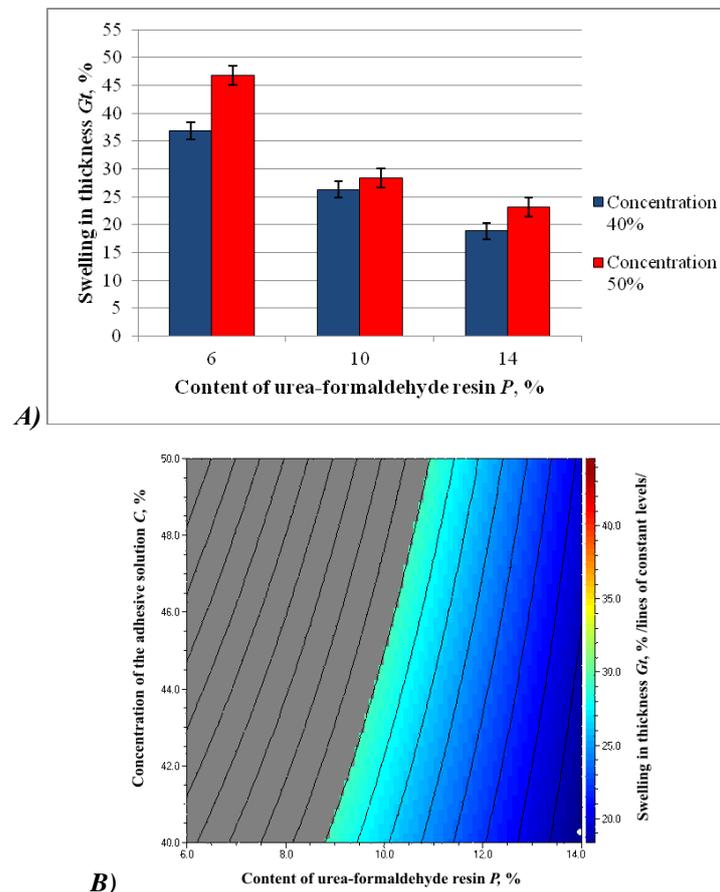
In graphic form, the variation of water absorption of boards as a result of the various urea-formaldehyde resin content and of the adhesive solution concentration is presented in Fig. 1.



**Figure 1: Variation of water absorption of MDF**  
**A) Effect of UFR content and concentration; B) Optimum water absorption value**

Under the investigation conditions, the MDF water absorption varies from 111% to 68%. I.e., improvement of the investigated indicator of approximately 1.66 times is observed. Therefore, the UFR content, at variation of 6% to 14%, and adhesive solution concentration, at variation of 40% to 50%, affect considerably the water absorption of boards. The effect of the two investigated factors is in opposite directions, with the effect of UFR content being about 3 times stronger than the effect of adhesive solution concentration. With the increase of the UFR content, considerable improvement of the water absorption of boards is observed, with the improvement being greater in case of increase of UFR content from 10% to 14%. Conversely, with the increase of the UFR content from 40% to 50%, deterioration in the MDF water absorption is observed. Therefore, the conclusion may be drawn that in spite of the higher moisture content in the wood-fibre mat, at adhesive solution concentration of 40%, considerably better UFR distribution is obtained at this concentration in comparison with concentration of 50%. The increased moisture content of the compactable material in all probability also leads to formation of additional hydrogen bonds in the board. It should be noted that the hot pressing factor is relatively high (60 s/mm) on account of which the gas-vapour mixtures that have formed have enough time to separate from the compactable material. Optimum (maximum) value of MDF water absorption of 68.67% is obtained at binder content of 14% and adhesive solution concentration of 41.45%.

In graphic form, the variation of swelling in thickness of boards as a result of the various urea-formaldehyde resin content and of the adhesive solution concentration is presented in Fig. 2.



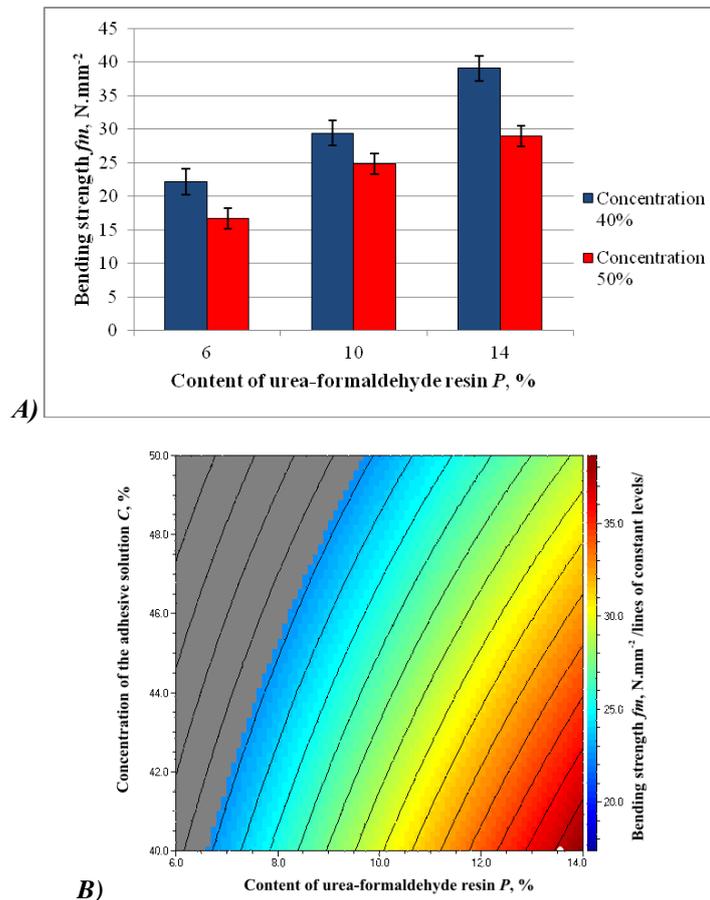
**Figure 2: Variation of swelling in thickness of MDF**

**A) Effect of UFR content and concentration on swelling in thickness; B) Optimum swelling in thickness value**

Under the experiment conditions, the swelling in thickness of MDF varies from 18.87% to 46.76%. With the increase of the UFR content, the swelling in thickness improves (decreases), whereas with the increase of the adhesive solution concentration the indicator deteriorates. In the investigated range, the intensity of the effect of the UFR content is 5 times greater than the intensity of the effect of the UFR concentration.

The limitation of the swelling in thickness pursuant to EN 622-5 (below 30%) is plotted on Fig. 2 B). MDF produced from wood of hard hardwood tree species at 40% concentration meet the standardization requirements at UFR content of at least 8.8%. At 50% adhesive solution concentration, the UFR content shall be at least 11%. The optimum, i.e. minimum value of swelling in thickness of 18.51% is obtained at 40.27% adhesive solution concentration and 13.96% UFR content.

Variation of bending strength of MDF as a result of the various urea-formaldehyde resin content and various adhesive solution concentration is presented in graphic form in Fig. 3.



**Figure 3: Variation of bending strength of MDF**

**A) Effect of UFR content and concentration on bending strength; B) Optimum bending strength value**

Under the experiment conditions, variation of UFR content at levels of 6%, 10% and 14% and variation of adhesive solution concentration at levels of 40% and 50%, the bending strength of MDF varies from 16.7 to 39  $\text{N}/\text{mm}^2$ . As with the remaining indicators, here also with the increase of the UFR content, the bending strength improves, and with increase of the concentration, it deteriorates. The intensity of the effect of the UFR content is 2 times greater than the intensity of the effect of the concentration. The requirement for bending strength of at least 23  $\text{N}/\text{mm}^2$ , for general purpose MDF and use in dry environment (EN 622-5) is plotted on Fig. 3 B). At adhesive solution concentration of 40%, this requirement is met already at UFR content of 6.5%, and at concentration of 50%, the necessary UFR content is 9%. The maximum value of the bending strength of 39  $\text{N}/\text{mm}^2$  is obtained at UFR content of 14% and concentration of 40%.

## CONCLUSIONS

As a result of the performed investigation, it has been established that, by means of control of the UFR content and the concentration of the introduced adhesive solution into the wood-fibre mass, MDF may be successfully produced entirely from wood of hard hardwood tree species.

With this type of wood raw material, it is recommended that the adhesive solution concentration be within up to 40%. The concentration increase leads to deterioration in the indicators of the boards. Therefore, at this relatively low concentration, better binder distribution is obtained, with the binder, unlike FB (fibreboards), not permeating into the wood interior, but remaining on the surface of the wood fibres. Most probably because of the increased moisture content of the compactable material, additional amount of hydrogen bonds among the fibrous elements also emerge. MDF produced with 14% UFR, but at 40% introduction concentration, have best indicators. Boards meeting the standardization requirements may be produced at minimum binder content of 8.8%.

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## Influence of climate oscillations on moisture content profiles of beech timber during initial stages of drying

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**Keywords:** wood drying, climate oscillations, beech, moisture content profiles

### ABSTRACT

This study aimed to determine the influence of equilibrium moisture content (EMC) oscillations on MC profiles of beech timber (38 x 120 x 500 mm) during drying in the climatic chamber. Two test runs with EMC oscillations (along with constant temperature) were followed by the equivalent test runs in constant climate: altogether, 4 drying runs were carried out. The amplitudes of EMC oscillations were ca. ±1.5% (absolute value) compared to the value in constant climate test, while the temperature in all cases was 35 °C. Among others, MC profile across thickness was periodically determined by slicing five lamellae and using the oven-dry method to determine their MC. Drying curves produced during constant and oscillation climate drying were similar and no statistical difference was found between average MC determined on the same day during constant and oscillation drying. In all cases it was found that MC difference between core and surface is lower (or at least not higher) in oscillating compared to constant climate conditions. This indicates that, if the positive influence of additional mechanosorptive creep (caused by EMC oscillations) on stresses in wood is taken into account, lower stresses in surface layers can be expected during oscillation drying. Further tests will be done to reveal the exact MC changes within surface layers of the boards. It is demonstrated that the climatic chamber is very stable in reaching and maintenance of air parameters during oscillation drying; it has practically no uncontrolled oscillations and will be used in further testing, along with the tests in semi-industrial and industrial kilns.

### INTRODUCTION

Improvement of conventional wood drying in terms of drying time and quality was the subject of many scientific studies in recent years. A number of research works are dedicated to the potential use of oscillating drying conditions. These conditions are already present in solar kilns where the periodic nature of the solar drying process ensures that drying stresses generated during the day are relieved during periods of cooling at night, when moisture condenses onto the surface of the timber (Herritsch et al. 2010). Artificially created oscillations during conventional drying were also in the focus of different studies (Salin 2003, Sackey et al. 2004, Milić and Kolin 2008).

Oscillating drying conditions provoke a continuous change of moisture content in the surface layers of boards which results in an enhanced mechanosorptive creep (Rémond et al. 2013, Salem et al. 2017). Its magnitude usually exceeds the amount of viscoelastic creep during conventional drying (Hanhijärvi 2007; Moutee et al. 2010). Although positive effects of oscillations on hardwood timber drying are reported in few studies (e.g. Milić et al. 2013, Langrish 2013), upscaling is a difficult task due to already present, uncontrolled oscillations in industrial kilns caused by the reversal of airflow (Riehl and Welling 2003) and dependent of stack depth, air velocity, wood species, current moisture content (MC).

The aim of this study was to determine the influence of EMC oscillations on moisture content profiles of beech timber during drying in the climatic chamber in which uncontrolled oscillations are diminished. The secondary goal was to test the stability of set parameters in the climatic chamber when working in an automatic oscillating regime.

## EXPERIMENTAL METHODS

Drying cycles were done in climatic chamber Kambič KK-105CH. The dry bulb temperature in the chamber is adjusted through a proportional-integral-derivative (PID) controlling electric heating elements and relative humidity of air is controlled through a water film, set at the dew point temperature, that flows at the bottom of the chamber. A fan for internal air circulation has speed control, and 1 m/s air velocity was used in this research. Six beech wood boards (38 x 120 x 500 mm) were dried in every drying cycle (Fig. 1). The initial length of the boards was 1050 mm, and then they were halved – one half was used for oscillation drying and other for constant (conventional) drying.



*Figure 1: Climatic chamber with beech boards*

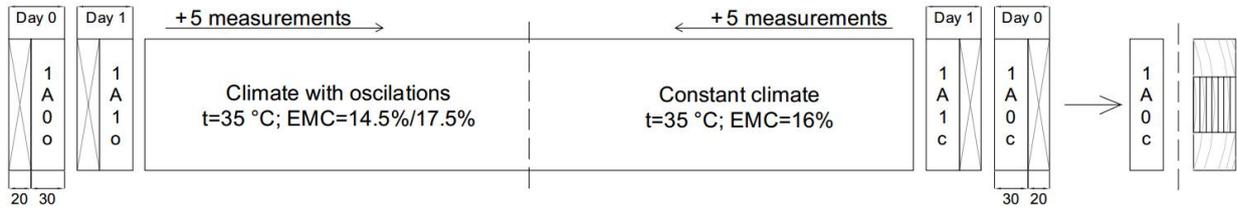
Altogether, 4 drying runs were carried out (Table 1). Every test run with EMC oscillations was followed by an equivalent test run in constant climate. The duration of test runs with milder climate (mark I) was 7 days, while harsher runs (with the mark II), both with and without oscillations, lasted 20 days. During “osc” drying runs, other halves of boards were put into the stretch foil to prevent loss of moisture.

*Table 1: Drying parameters in four test runs (beech 38 mm)*

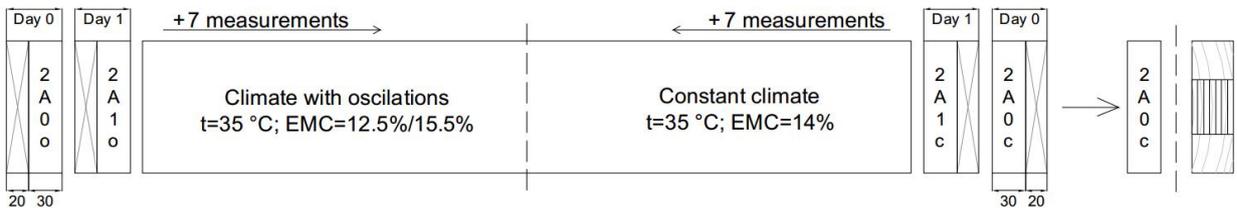
Test run	Temperature [°C]	EMC [%]	Frequency [h]	Duration [days]
I osc	35	14.5/17.5	3	7
I con	35	16	-	7
II osc	35	12.5/15.5	3	20
II con	35	14	-	20

Among others, MC profile across thickness was determined before, during and after drying by slicing five lamellae (Fig. 2). Then the oven-dry method was used to determine their MC. The procedure was repeated 7 times (every day) for test runs I and 9 times (every 2 days during the first 9 days and every 4 days later on) for test runs II. After each cutting aluminium foil was placed at the board end to avoid intensive drying of the ends.

Test run I



Test run II



**Figure 2: Scheme of cutting samples and lamellae**

Moisture content difference across thickness for every board ( $\Delta MC_i$ ) was calculated (Eq 1.) as the difference between moisture content in the centre of the board ( $MC_c$ ) and the mean moisture content in the board surface ( $MC_{s1}, MC_{s2}$ ):

$$\Delta MC_i = MC_c - \frac{MC_{s1} + MC_{s2}}{2} [\%] \quad (1)$$

The mean moisture content difference was then calculated by the formula:

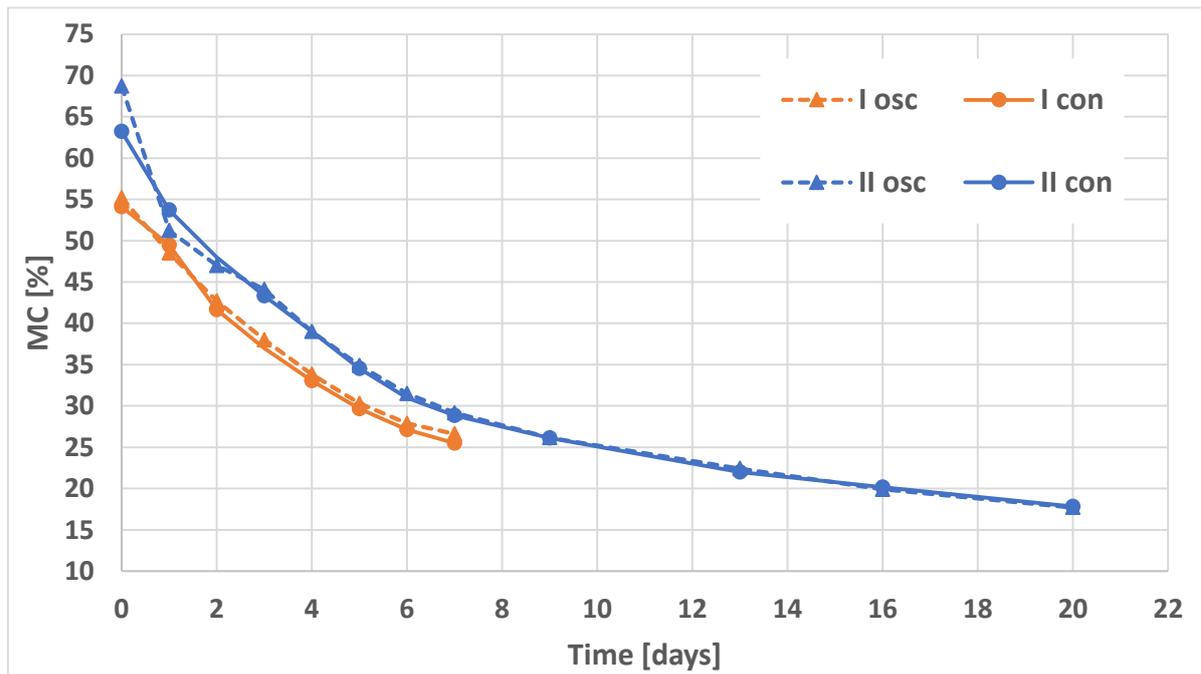
$$\Delta MC = \frac{\sum \Delta MC_i}{6} [\%] \quad (2)$$

The current MC of every board was calculated based on the sum of the mass of all 5 lamellae.

## RESULTS AND DISCUSSION

Conditions in the climate chamber were very stable during all test runs. Stability was high both for temperature ( $\pm 0.05$  °C) and relative humidity ( $\pm 0.2\%$ ) and the transition between oscillations phases was very fast – approx. 1 minute was needed to reach a new relative humidity value after the phase change. No visual defects (surface checks, deformations, discolourations) were found on boards in any test runs. This result, especially in the case of test runs II (with lower EMC), demonstrates the potential to use lower EMC values in combination with relatively low air velocity during initial stages of conventional drying in the industry.

Drying curves produced during constant and oscillation climate drying were similar (Fig. 3). No statistical difference was found between average MC determined on the same day during constant and oscillation drying. In the test runs II initial MC was higher compared to test runs I, which was also reflected in higher MC during the first 7 days of drying. After this period, the average MC was between 25% and 30% in all test runs. Drying rate below 30% MC in test runs II was relatively low due to unchanged EMC (average 14%, which is high for this stage of drying). After 20 days average MC was the same (approx. 18%) both in the test run with and without oscillations. Variation in MC between boards was very low (min. 17.2%, max. 18.2%).



**Figure 3: Average moisture content of boards during oscillating and constant drying conditions**

Although the average MC of the boards was similar in oscillation and constant climate, EMC oscillations clearly influenced MC in a thin surface layer of boards which was registered even with relatively thick surface lamellae (approx. 8 mm). This resulted in a lower MC gradient across the thickness (Fig. 4) compared to results in constant climate, especially during test run I (milder conditions).

In both cases, initial  $\Delta$ MC was lower for constant compared to oscillation runs which was a result of moisture redistribution during staying in foil (during test run I that period was 7 days, and for a test run II 20 days – therefore initial  $\Delta$ MC for a run “II con” was about 0%).

Initial  $\Delta$ MC was higher in test runs I compared to test runs II as a result of lower initial MC, i.e. few days of air drying of these boards. Part of free water already evaporated from surface layers and caused lower initial MC, but also higher MC gradient during the first days of drying in the climatic chamber. The maximal average value of  $\Delta$ MC during drying for “I osc” was 5.3% (at the end of the process), while for “I con” that was 5.8% (after 4 days of drying). For individual boards with higher initial MC (above 60%) the difference between oscillating and constant climate was even higher. Maximal  $\Delta$ MC value for the individual board was 6.5% for an oscillating and 9.0% for a constant climate. With this much higher  $\Delta$ MC reached in a constant climate, higher internal stresses can be expected.

Test runs II, where the boards had a higher amount of free water in surface layers, started with low MC gradient which gradually raised, and maximum values were reached between 8<sup>th</sup> and 12<sup>th</sup> day of drying ( $\Delta$ MC approx. 7%). Here, for individual boards,  $\Delta$ MC was a maximum of 7.8% for oscillation and 9.2% for the constant climate.

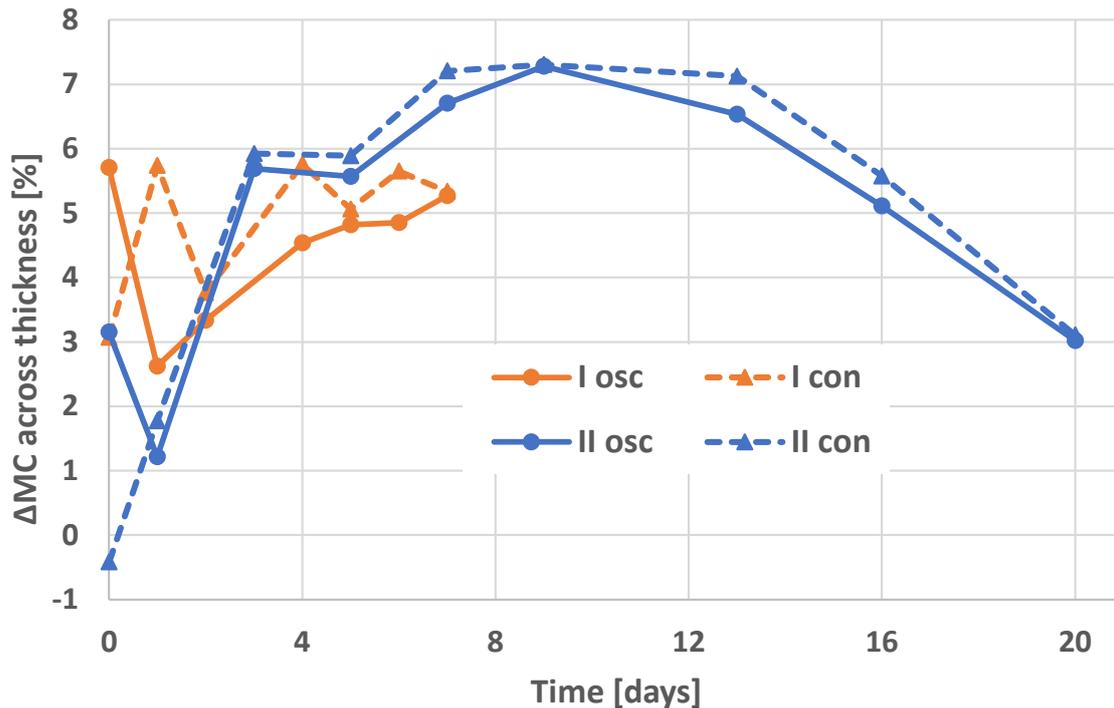


Figure 4: Moisture content difference across thickness during oscillating and constant drying conditions

In all cases it is clear that  $\Delta MC$  is lower (or at least not higher) in oscillating compared to constant climate conditions. Mechanosorptive strain was not measured, but it is well known from previous studies (e.g. Langrish 2013; Rémond et al. 2013) that changing MC in surface layers, due to EMC oscillations, is causing additional mechanosorptive strain. This additional strain reduces overall instantaneous strains in surface layers of the boards. In total, lower stresses can be expected in surface layers during oscillating drying conditions as compared to the conventional process.

## CONCLUSIONS

In this study we have tested the influence of EMC oscillations (along with unchanged temperature) during initial stages of beech timber drying on MC profiles across the thickness. It was found that the drying curves produced during constant and oscillation climate drying were very similar which confirmed that EMC oscillations have no influence on average MC of the boards. However, MC difference between center and surface was somewhat lower in tests with oscillations as compared to tests with constant climate conditions. If the positive influence of additional mechanosorptive creep on stresses in wood is taken into account, lower stresses in surface layers can be expected during the initial stages of oscillation drying.

Further tests will involve different amplitudes and frequencies of oscillations, but also 2-steps drying schedules in order to evaluate the influence during later stages of drying. Furthermore, more lamellae (at least 7) will be cut from samples to reveal more accurately the influence of oscillations on surface moisture content.

Although testing of EMC oscillations in semi-industrial and industrial kilns will continue, drying in the programmable climatic chamber has been shown to be a useful method to avoid effects of uncontrolled oscillations during experiments.

## ACKNOWLEDGMENTS

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## Wood research of the tree-of-heaven (*Ailanthus altissima*) at the Institute of Wood Science in Sopron

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**Keywords:** hardwood, mechanical properties, heat treatment, dimensional stability, density, energetic usage, colour measurement

### ABSTRACT

The technical properties of the wood of tree-of-heaven (*Ailanthus Altissima*) are significantly determined by the place of growth. It has an air-dry density of 600-700 kg/m<sup>3</sup> and average mechanical values like modulus of rupture of 94.83 MPa, modulus of elasticity of 12.20 GPa, shear strength of 10.66 MPa, compressive strength of 64.49 MPa and end-grain hardness of 35.70 MPa. These are test results for wood samples came from trees grown in poor quality sites. Wood modifications are worthwhile to expand the possibilities of using *Ailanthus*, an excellent interior design and furniture raw material that is similar to ash in many respects, but not suitable for outer use because of its weak natural durability. The dry heat treatment has been proved as an alternative method to reach higher resistance class against basidiomycetes, thus the service life of the wooden constructions and products can be prolonged in case of outdoor applications, too. Using linseed oil, heat treatment was performed at a temperature of 200 °C for 6 hours. The samples were differentiated into three groups according to the moisture content before heat treatment: 0%, ~12% and green state. For green state samples, the cracks became large and very frequent. The cracks occurred mostly next to the rays and along the middle lamellae.

### INTRODUCTION

The tree-of-heaven (*Ailanthus Altissima*) is native in central and north-eastern China and Korea but it also can be found in large quantities from East Asia to Northern Australia. It got into Europe in 1751 as park tree. It has been planted in Hungary since about 1820. In Hungary slower-growing, more winter-resistant varieties (*Ailanthus altissima* or *Ailanthus glandulosa*) has become widespread. It is mainly the tree of the plains and hills. It is warm-loving, sensitive to early frosts and to the cold weather in winter. It also tolerates extreme (e.g. dry) areas but provides good growth and good wood quality with great dimensions on well-managed, airy, humus-rich soils. It requires extremely much light (Gyuricsek 2015).

Summarizing the results of several researchers, today due to EU directives *Ailanthus* has been blacklisted in Hungary as a non-native tree species because its interspecific competition ability, its negative effects on ecosystem functioning, its large current distribution and its high reproductive and spread capacity. It is also spreading these days, monopolizes resources and having a facilitation effect on climate change. According to the Hungarian classification, it is an invasive transformative species, and it is also considered an invasive species in all the surrounding countries. According to both EASIN and DAISIE classifications, it is considered as high impact wood species, while EPPO classified it as invasive alien plant. It is both on the EU-IAS and Widespread IAT&SS lists but is not among the 100 of the worst invasive alien species in Europe and in the world (Bartha 2020).

*Ailanthus* grows extremely fast in its early years, reaching a height of 20-25 m and a diameter of 0.3-0.5 m at the age of 35-40 years. Easy to plant and germinates excellently. Unfortunately, often suffers storm break. Its leaves and young sprits have an unpleasant odour, the bark is also very bitter, thus, biotic pests avoid it. Its leaves have large dimensions (40-60 cm long) and each leaf consists of 13-25 short-stemmed, ovoid, lanceolate-shape small leaves. Its bark is smooth, gray, with elongated white cracks on older trees. *Ailanthus* blossoms after robinia (June-July), a good nectar-source for honey (Molnár and Bariska 2005, Fehér and Komán 2014). Although it has been declared an undesirable tree species in Europe, the utilization of

existing and – in the future – less and less, but still arising amount of wood is an important task from an environmental, forestry and national economic point of view.

The literature about the modification of less utilized wood species, such as *Ailanthus* is very poor. Although, some examples can be found, e.g. that the veneers of *Ailanthus* modified by urea-formaldehyde pre-polymer and compression drying can improve its properties (dimensional stability, bending strength, tensile strength parallel to the grain). Its hygroscopicity could be reduced significantly and its thermal stability was enhanced, too (Miao et al. 2014). It shows a strong similarity to European ash (*Fraxinus excelsior*) in appearance and physical and mechanical properties (Brandner and Schickhofer 2013, Gyuricsek et al. 2014).

Due to the frequent occurrence and growth characteristics of *Ailanthus*, the study of its utilization for the wood industry and energy purposes can be considered actual. Therefore, more scientists have dealt with *Ailanthus* to explore the optimal and modern utilization possibilities of that wood, based on its technical properties. The assessment of the quality of wood varies. Alden (1995) showed very modest values for the density of *Ailanthus* (0.54-0.62 g/cm<sup>3</sup>), but its elastic properties obtained outstanding results compared to the density. Its modulus of elasticity is 10.48 GPa and its modulus of rupture is 81.36 MPa. It is considered to be a particularly high-quality wood based on its physical and mechanical properties (Panayotov et al. 2010). The *Ailanthus* is very similar to ash in terms of the tissue and anatomical structure of its wood. Thus, it is advisable to combine its study with measurements of the properties of ash (Fehér and Komán 2014). *Ailanthus* has a low durability outdoors. It is ring-porous, in the latewood the vessels form tangential lines. Spiral cell wall thickening can be observed on the vessel walls. Its wood rays are clearly visible to the naked eye, they can be observed as 1 mm high silky spots on the radial section. The fibers of *Ailanthus* are 1.2-1.3 mm long. The sapwood is wide and yellowish and the heartwood has a greyish-orange colour, hard to differentiate them (Molnár 2004, Varga 1995).

There may be many possibilities to use *Ailanthus* in the future in addition to furniture and interior design, a good example is its application in laminated veneer lumber (LVL). An attempt was made to improve the elastic properties of poplar LVL by Vilpponen et al. (2014a and 2014b). Of the outer layers, 2 veneers were replaced with *Ailanthus*. The non-destructive modulus of elasticity of the LVL specimens was much greater than expected as a result of the *Ailanthus* reinforcement layers. *Ailanthus* veneers, which are otherwise of lesser importance in industry and are considered to be of lower value, are particularly suitable for improving the properties of LVL made from poplar veneer. The use of *Ailanthus* reinforcing layers provided very moderate, but still statistically significant increase in modulus of rupture.

This study summarizes the wood properties of *Ailanthus*, with a particular focus on researches conducted by the Institute of Wood Science at the University of Sopron in the recent decades. The published study is a collection of results achieved by our instructors and researchers. During the past decades several BSc and MSc theses have dealt with this wood species. In the chapter Results and Discussion, we try to present the results and correlations. For this reason, it is unnecessary to specifically discuss the wood properties of *Ailanthus* here, in chapter Introduction.

## EXPERIMENTAL METHODS

The researches presented in the Results and Discussion chapter was prepared in accordance with the relevant Hungarian and international standards (e.g. MSZ 6786-3:1988, MSZ 6786-5:1976, MSZ 6786-6:1977, MSZ 6786-8:1977, MSZ 6786-15:1984). The results are therefore repeatable. For details regarding the test conditions or other questions, it is worth reviewing the cited studies, which contain much deeper and more detailed information. They can be found in English or Hungarian, depending on the source of publication.

## RESULTS AND DISCUSSION

Testing some properties of a young *Ailanthus*, Varga (1995) found that its tensile strength was averagely 199.11 MPa, which is a remarkably high result compared to the tensile strength of ash (165 MPa). This may have been due to high growing rate during the first years, thus wider annual rings and less vessels (ring pores), but higher share of thick-walled fibers responsible for strength. The mean value of the tensile

modulus of elasticity from 60 measurements was 10.92 GPa. The coefficient of variation of the results are 11.11% and 23.95%, respectively. The moisture content of the samples was higher than the standard, thus, in each case the values were converted to 12% moisture content. Compressive strength values showed an increasing trend with increasing density. The average density of the tested samples was  $0.61 \pm 0.03 \text{ g/cm}^3$ , with an associated compressive strength of 69.49 MPa and a coefficient of variation of 16.71%. The Krippel-Pallay hardness, which gives very reliable hardness results, was 35.7 MPa on the end-grain, 14.91 MPa in the tangential direction and 11.43 MPa in the radial direction. The coefficient of variations are 15.83%, 17.51%, and 24.21%, respectively. The hardness of the samples also increased in line with their density ( $0.63 \pm 0.04 \text{ g/cm}^3$ ).

After the examination of 75 samples, Várhegyi (1995) found an average density of  $0.61 \text{ g/cm}^3$  with a confidence interval of 0.01 at 5% significance level. The average moisture contents ranged from 14% to 17%, the results are converted to a moisture content of 12%. The modulus of rupture was  $94.83 \pm 2.90 \text{ MPa}$ , while the bending modulus of elasticity was  $12.20 \pm 0.20 \text{ GPa}$ . The shear strength parallel to the grain was  $10.66 \pm 0.51 \text{ MPa}$ , the nail-withdrawal resistance 2 hours after nailing was  $64.36 \pm 0.84 \text{ N/mm}$  in the radial direction and  $82.33 \pm 1.62 \text{ N/mm}$  in the tangential direction. One week after nailing, these values became  $59.27 \pm 1.54 \text{ N/mm}$  and  $69.84 \pm 1.14 \text{ N/mm}$ , respectively. The large variation over time was partly due a decrease in moisture content.

Because of their similarities, the properties of *Ailanthus* have been compared to European ash (*Fraxinus excelsior*) in many studies. As an example, Vargáné and Fehér (1996) found that the strength properties of *Ailanthus* allow a wide range of applications. The strength and hardness values found by Varga (1995) and Várhegyi (1995) are similar to ash (Table 3), the modulus of rupture remains slightly below, but the compressive strength is higher. All its strength values exceed the values of oak, alder, chestnut, elm and characterize a flexible material that can be used as a load-bearing element. Due to its beautiful texture and colour, it is expedient to use it in areas of application where – in addition to strength properties – aesthetics also play a role: interior design, wall covering, parquet production, marquetry.

**Table 3: Comparison of *Ailanthus* and European ash test results (source: Vargáné and Fehér 1996)**

	<i>Ailanthus</i>	Ash
Density [ $\text{g/cm}^3$ ]	0.61	0.69
Shear strength [MPa]	10.66	12.00
Brinell hardness, end-grain [MPa]	-	65.00
Krippel-Pallay hardness, end-grain [MPa]	35.70	45.20
Modulus of Rupture [MPa]	94.83	120.00
Modulus of Elasticity [GPa]	12.20	13.40
Compressive strength [MPa]	64.49	52.00

In the study of Fehér and Komán (2014) the null hypothesis was that the technical properties of *Ailanthus* do not differ from the European ash based on its similar annual ring structure and anatomical structure. They found that the samples examined by Varga (1995) and Várhegyi (1995) came from a lower quality site. Aside from that density tests gave lower values for *Ailanthus* compared to ash ( $0.76 \text{ g/cm}^3$ ), especially for the samples came from the lower-quality site ( $0.60 \text{ g/cm}^3$ ), some strength characteristics do not show large differences (Table 1). Although the quality of the site apparently results in a significant change in the density, the density of the samples from a better site already reaches the limit of  $0.70 \text{ g/cm}^3$ . Accordingly, the hardness values also show nearly similar values.

**Table 1: Results of the density and hardness tests (12% MC) (source: Fehér and Komán 2014)**

	Density [ $\text{g/cm}^3$ ]	Brinell-Mörath hardness, end-grain [MPa]	Brinell-Mörath hardness, side [MPa]	Krippel-Pallay hardness, end-grain [MPa]
<i>Ailanthus</i> , poor site	0.60	-	-	35.70
<i>Fraxinus</i> , average site	0.76	55.48	33.45	45.20
<i>Ailanthus</i> , average site	0.71	68.28	28.66	-

Strength tests also gave similar results as density and hardness tests. Only in the impact strength and modulus of rupture tests can clearly be detected significant difference in favour of European ash (Table 2).

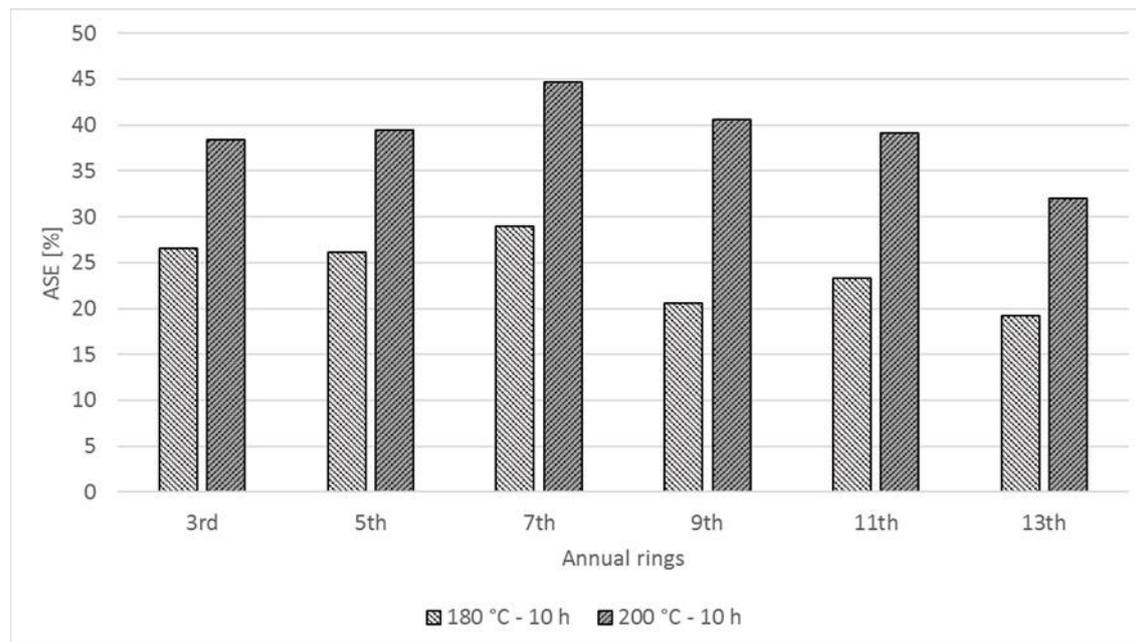
For other tests (shear strength, compressive strength, modulus of elasticity), such a clear conclusion can not be drawn, because depending on the site's location the results reach or possibly exceed the strength of ash.

**Table 2: Results of the mechanical tests (12% MC) (source: Fehér and Komán 2014)**

	Shear strength [MPa]	Compressive strength [MPa]	Impact strength [J/cm <sup>2</sup> ]	Modulus of rupture [MPa]	Modulus of elasticity [GPa]
<i>Ailanthus</i> , poor site	10.66	64.49	-	94.83	12.20
<i>Fraxinus</i> , average site	12.27	60.14	11.00	114.53	12.36
<i>Ailanthus</i> , average site	16.73	45.54	7.09	94.83	10.21

The average calorific value of the samples from the weaker site was 19.43 MJ/kg. Compared to the data of the literature, the calorific value is practically the same. In contrast, there is a significant difference between the ash contents: the value found in the literature (2.22%) is almost double that of the values of our own investigations (1.14%). The significant difference is due to the difference in the amount of mineral materials in the wood, moreover, the proportion of the bark (Fehér and Komán 2014).

After analysing the results of *Ailanthus*, we could prove, that the average equilibrium moisture content (T=20 °C and RH=65%) of native samples was 13.6 %, while after dry heat treatment at 180 °C it was significantly lower (7.2%). For heat treatment at 200 °C the samples showed 4.8% moisture content in average at normal climate. The differences between the moisture content values of the selected annual rings were not significant in comparison to each other. It was determined that the dry heat treatment has the same effect on the whole wood tissue, independently to the annual rings' positions. For radial swelling, a significant decrease was determined in the direction to sapwood due to the treatments. The tangential anti-swelling efficiency achieved ca. 19-26% for the samples heat-treated at 180 °C and 32-44% for the samples heat-treated at 200 °C (Fig. 1) (Gyuricsek et al. 2014, Németh et al. 2020)



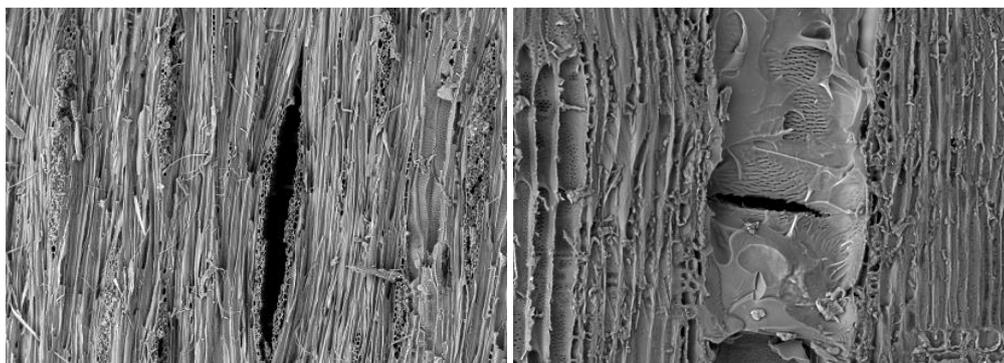
**Figure 1: The improvement of ASE in tangential direction of Ailanthus (source: Németh et al. 2020)**

The density showed also low deviation from the 3rd towards the 13th annual ring. The average air-dry density of the native wood specimens was 0.64 g/cm<sup>3</sup>. After the treatments at 180 °C and 200 °C the average densities of the treated *Ailanthus* samples varied averagely between 0.60 g/cm<sup>3</sup> and 0.59 g/cm<sup>3</sup>. The density of the annual rings increased with the age of the tree with a total difference of 0.03-0.07 g/cm<sup>3</sup>. The density seems to be only increased from inside up to the 9th annual ring. This may be related to the juvenile wood's boundary in the xylem between the 9th and 11th annual rings. The CIE Lab measurements verified that dry heat treatments have a similar effect on the *Ailanthus* wood samples than on the other wood species. The lightness (L\*) of the untreated specimens was 80.13, which decreased after heat treatment at 180 °C temperature to 59.15 and at 200 °C temperature to 38.60. The average value of red colour component (a\*) of native specimens was 3.67, which changed to 9.99 and 11.75, respectively, so the samples turned to a reddish colour. The average value of yellow colour component (b\*) increased from 23.25 to 28.27 and 29.71, respectively (Gyuricsek et al. 2014).

The oil heat treatments of *Ailanthus* were performed in linseed oil at the temperature of 200 °C during 2, 4 and 6 hours. For samples treated with green state initial moisture content, the cracks could be seen well with the naked eye (Fig. 2.) The cracks occurred almost only in radial direction. Using SEM imaging, most of the micro-cracks could be observed next to the rays or in the rays between the single ray cells (Fig. 3.). As the moisture leaves the wood in the form of steam during heat treatment, the steam pressure increases rapidly in the lumens. The reason for the high amount of the cracks in this region is the thin and weak cell wall of the ray parenchymas, which collapse easier than the libriform fibers. Several cracks were continued from the rays into the ground tissue (libriform fibers) of the wood as well. In the vessels, only a few and small cracks occurred as a result of the treatment. *Ailanthus* has large and open vessels without tyloses, thus, the steam formed during heat treatment in the cell walls and lumens can leave the wood tissue easily. Therefore, the stresses induced by the increased steam pressure are lower in the vessels as well. On the other hand, the collapses occurred often between the cells along the middle lamellae, which is also a weak point in the wood structure (Bak et al. 2009, Németh et al. 2020).



**Figure 2: Radial cracks and deformations on the cross section of *Ailanthus* (source: Bak and Németh 2015)**



**Figure 3: SEM images from cracks of *Ailanthus* tangential surface (left) and radial surface (right) (source: Bak and Németh 2015)**

## CONCLUSIONS

Although European countries are trying to limit the spread of tree-of-heaven (*Ailanthus altissima*) due to its invasive habitat, its wood has been the subject of numerous research projects across Europe. Utilization of its timber is still in its infancy. Its durability is poor, so its applicability is limited. Therefore, technical researches focus mostly on the properties of wood in the context of optimal and modern utilization. This study introduces and summarizes the researches of the wood of *Ailanthus* conducted in Sopron in the last decade.

In a lower quality growth site, it has only a medium quality timber with a density of about 600 kg/m<sup>3</sup>. Hence, its density grown under better conditions exceeds 700 kg/m<sup>3</sup>. The properties of ash wood may be reached or possibly even exceeded. Exceptions are the elastic properties, modulus of rupture and modulus of elasticity. Thus, *Ailanthus* cannot be applied in wooden constructions, areas where higher demands appear. But its indoor use is unlimited: excellent raw material for wall coverings, stairs, furniture, etc. Care must be taken to ensure that the green wood does not attacked by blue stain fungi, which significantly impairs its aesthetic properties. Due to the extraordinarily high dry matter production, *Ailanthus* has already been in the focus of energetic wood plantations. Its calorific value exceeds 19 000 MJ/kg, while its ash content is very low, just slightly over 1%.

As a result of heat treatment under atmospheric conditions with a duration of 10 hours, the equilibrium moisture content of untreated, treated at 180 °C and treated at 200 °C *Ailanthus* was 13.6%, 7.2 % and 4.8%, respectively, after conditioning at 20 °C / 65%. The differences of the equilibrium moisture content between the annual rings were not significant, but correlation could be found between the anti-swelling efficiency and the position of annual rings. The tangential anti-swelling efficiency achieved ca. 19-26% for the treatment at 180 °C and 32-44% for the treatment at 200 °C. The average density decreased from 641 kg/m<sup>3</sup> to 604 kg/m<sup>3</sup> and 591 kg/m<sup>3</sup>, respectively. According to CIE Lab colour measurement, the lightness of the untreated samples was 80.13, which decreased after treatments at 180 °C to 59.15 and at 200 °C to 38.60. The average value of red colour component rised from 3.67 to 9.99 and to 11.75, respectively, while the yellow colour component increased from 23.25 to 28.27 and to 29.71, respectively.

Using linseed oil at a temperature of 200 °C for 6 hours, the role of the initial moisture content of the samples was clear, as higher moisture content caused larger cracks and their number increased, too. In case of 0% initial moisture content only a few and very small damages were observed, and a little more and larger cracks in case of ~12% initial moisture content. For green state samples, the cracks became large and very frequent. The cracks occurred mostly next to the rays and along the middle lamellae.

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## Fundamental aspects of gluing technology: delamination test of window friezes

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### ABSTRACT

Water based poly(vinyl-acetate) – PVAc dispersions are worldwide known due to their advantageous properties, such as low VOC content, high chemical stability and bond strength, fast setting speed, and favourable price (Chiozza and Pizzo 2016). Most significant consumers are among wood, paper, and textile industry and used in the most various applications (such as tobacco processing, water emulsion paints, wood and paper adhesive formulation) (Chiozza et al. 2018). Water-based wood adhesives can be classified into four durability gradings according DIN EN 204 (2017) and DIN EN 205 (2017) standards (running from D1 to D4). These tests are carried out using beech wood specimens and are based on the different water-resistance of the adhesives. However, these measurements can not guarantee the applicability of the adhesives in the various applications, simply because the gluing process is strongly influenced by different factors, such as temperature, humidity or the materials used. On top of that, the demand for low cost, easily feasible tests is increasing, so that the different companies can find the best possible adhesive for their specific application. Window friezes are made by block glueing and often glued with two-component D4 adhesives due to the outdoor application. Oak and beech starting materials are often preferred over pine tree. This is because the high resin content of the pine often reduces the bond strength and stability thanks to its hydrophobicity. In this paper, a delamination test procedure is introduced, which can be easily done in industrial environment on window friezes. The results on different wood species and the effect of using modified adhesives are presented.

### INTRODUCTION

The most widely used water-dispersed material in glueing process is PVAc. PVAc is produced by radical polymerization of vinyl acetate. It is a biodegradable, thermoplastic polymer that has an excellent chemical resistance and no toxic effects on human health (Lutz et al. 2012). Copolymerization of vinyl acetate with other monomers (such as ethylene) increases the possibility of various applications (adhesives for paper and tobacco industry). Dispersing the solid product in water with stabilizers (such as polyvinyl alcohol) and other materials gives aqueous wood adhesives. Depending on the condition of polymerisation and other added additives (crosslinker and hardener), the formulation can result in glues of different water durability grades (from D1 to D4) according DIN EN 204 and 205.

Unfortunately, these standards can only qualify the water resistance properties of the glue, and not the gluing process that is strongly influenced by many different parameters, such as temperature, applied quantity, pressing time and pressure, open time, air humidity, water content, as well as quality and annual ring of the wood samples. The minimum film forming temperature ( MFFT ) is a very important property of PVAc glues. MFFT is the lowest temperature at which the adhesive will uniformly coalesce and form a coherent, continuous film upon drying. In that manner, the temperature of the samples as well as the temperature of the room where the gluing process took place are very important. Open time is the time between adhesive application and pressure application. This parameter is also very important and depends on many factor. The pressure of the pressing has to be precisely controlled as well. Too high pressure forces

the adhesive out of the gluing plane, which indirectly results in a decrease of the quantity applied. Too low pressure might not be enough to complete the process.

Therefore, *easily feasible* tests are needed that *represent the circumstances of use*. In the followings, a delamination protocol on Accoya® and oak window friezes glued with different adhesives will be introduced. This test comes from one of our main partners who always checks the quality of their basic commodity to maintain client satisfaction.

## EXPERIMENTAL METHODS

Accoya® and oak tree samples were examined according to the following protocol (Table 1).

**Table 1: Complete protocol of delamination test on window friezes.**

STEP NUMBER	STORAGE CONDITIONS
1.	Before gluing: 7-day storage at standard ambient conditions*
2.	Gluing process
3.	After gluing: 7-day storage at standard ambient conditions*
4.	3-hour storage of wood samples in cold water (20 ± 1 °C)
5.	3-hour storage of wood samples in hot water (60 ± 1 °C)
6.	18-hour storage of wood samples in cold water (20 ± 1 °C)
7.	72-hour storage at standard ambient conditions*

\*Standard ambient conditions: 23,0 °C / 50 % relative humidity.

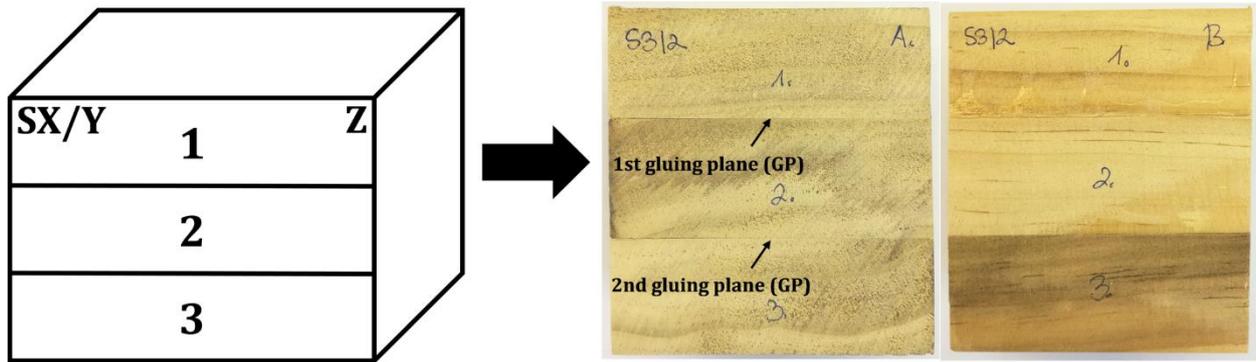
Before step Number 1, staves of Accoya® and oak tree were planed. Prior gluing, the surface of the staves were sanded with a Bosch® PSS 250 AE low vibration machine using abrasive paper of grain size P100 complying with FEPA 43-1984. Staves were glued (step Number 2) via lamination with different adhesive products from our portfolio (Table 2). According to the method, three individual wood pieces were glued to each other.

**Table 2: Materials used in experiments.**

MATERIAL NUMBER	MATERIAL
1.	Technobond 3000
2.	100 % Technobond 3000 + 5 % D3D4 Additive
3.	Technobond 3016
4.	100 % Technobond 4201 (A) + 5 % Additive
5.	100 % Technobond 4201 (B) + 5 % Additive

Technobond 3000 and Technobond 4201 are our basic D3 and two component D4, water based PVAc product. D3D4 Additive is a mixture of different polyisocyanate, that increases the water resistance (from D3 to D4 durability grade). Technobond 3016 is a D3, high water-resistance formulation of Technobond 3000 with post-added hardener and polyvinyl alcohol with high degree of hydrolysis. Technobond 4201 without Additive is a D2 class adhesive. Material Number 4 and 5 and used Additive are the same, but made with unmodified 2 component D4 dispersions from different suppliers (A and B).

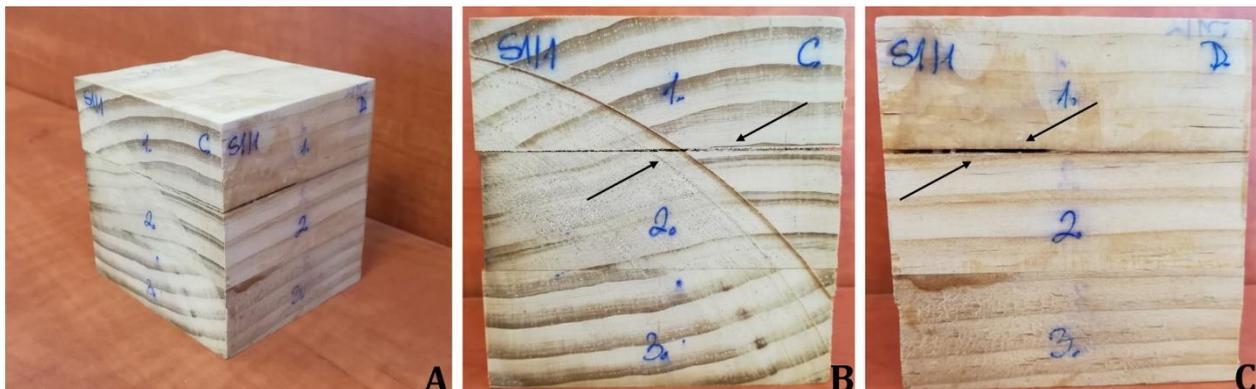
Gluing parameters were the following: two sided application, applied quantity on each side was 120 g/m<sup>2</sup>. After applying the adhesive, surfaces were come into contact as soon as possible and pressed. Pressing time and pressure were 2 hours and 8 kg/cm<sup>2</sup>, respectively, at room temperature in a hydraulic press. After gluing, the staves were cut into 3 identical samples with the parameters of L × W × H : 8,0 × 7,0 × 7,5 cm (Figure 1).



**Figure 1:** Schematic illustration of identical wood samples, where *S* means the Sample word, *X* is the adhesive sample number from Table 2. (1 – 5), *Y* means number of the equal wood sample (1 – 3), *Z* means the side of the wood specimen that we are talking about (A – D). Wood pieces number 1-2 and 2-3 define the first and second gluing plane (GP).

## RESULTS AND DISCUSSION

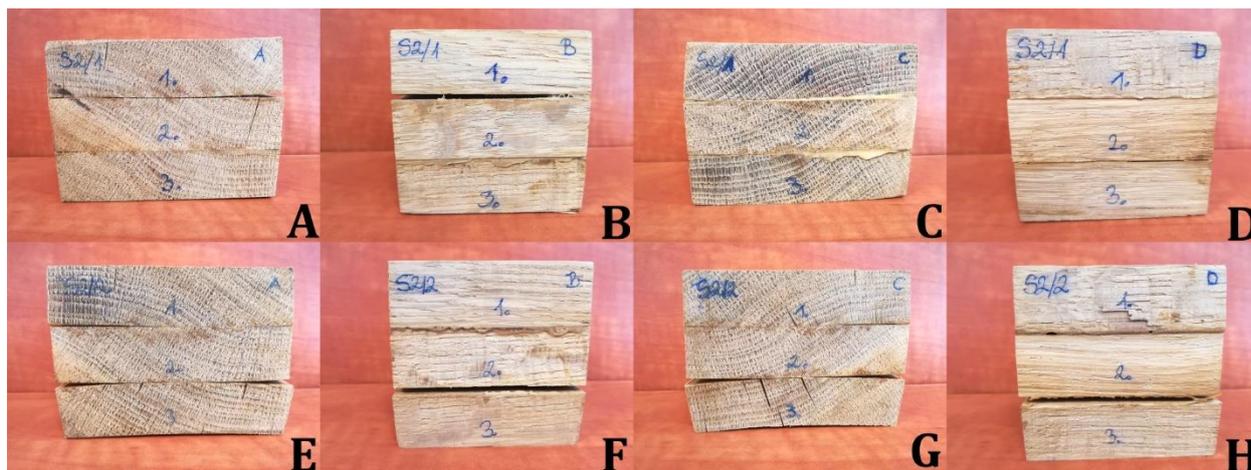
At the end of the delamination test (after step Number 7), wood samples were compared with respect to the gluing plane. Openings occurred on Accoya® samples only in the case of Technobond 3000 product (Figure 2).



**Figure 2:** Accoya® wood sample glued with Technobond 3000 (A) and openings on side C (B) and D (C) after delamination test. Openings are marked with black arrows.

Using water-based adhesives in gluing pine wood, high resin content of wood is always a significant problem. Resin contains mainly hydrophobic components. When adhesive is applied on the wood samples, the first step of the gluing process is the wetting of the wood surface. The second step is the diffusion of the adhesive components and water into the well-defined microstructure of pine (cell cavities). Too high resin content, so hydrophobicity inhibit the second step what leads decreasing the bonding strength. For this reason, solvent-based adhesives are often preferred over water-based products (despite the environmental problem concerning organic solvents). With isocyanate-based crosslinker and hardener, prominent gluing strength can be achieved. In that way, two components D4 adhesives can also be applicable.

Openings also occurred on oak wood, but only in the case of Technobond 3000 + D3D4 Additive (Figure 3).



**Figure 3: Pictures of different side of oak samples after delamination test glued with Technobond 3000 + D3D4 Additiv product. On A – B and E – H pictures, significant opening can be observed in the first and second gluing plane as well.**

Isocyanate crosslinkers and hardener are very reactive, which upon application results in not only the crosslinking of the chain molecules in the PVAc dispersion, but also a chemical modification of the wood surface and material. This modification can lead to improper wetting properties because of appearance of less polar functional group in the chemical structure of the wood. On the other hand, annual ring structure of wood samples are not so symmetrical, so in this case it could be also the reason of insufficient gluing.

## CONCLUSION

Generally, the delamination test described in this study can be successfully applied to compare adhesives of different kinds. For Accoya® wood most of our products passed the detailed test, except Technobond 3000 product that can be explained by the high amount of hydrophobic resin in wood material. Gluing oak with our products also gave good results, but in the case of Technobond 3000 + D3D4 Additive, significant opening can be observed on all samples by the end of the test. Our further plan is to test these products on beech samples.

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## Preliminary researching the heating intensity of wood as an effect of microwave radiation

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**Keywords:** *Pinus sylvestris*, microwave radiation, warming up wood, fly larvae, *Hylotrupes bajulus*

### ABSTRACT

Microwave treatment is a technology also used in wood protection, including treating deciduous and pine wood. Wood boring insects and wood boring mould can be eliminated by treating the wood with microwave. In our experiment we examine the heating of different types of wood (beech, oak, pine and spruce) using a MWG-1000-A-1 type generator, with a pyramid shaped applicator. First we heated up lamella made of pine (*Pinus sylvestris*) sapwood and measured the heating intensity of the lamellas in relation of the cross section of the wood. For our experiment we modelled a 150mm thick solid wood, placing on each other 6 pieces of lamella, each conditioned in normal climate and measuring 25mm in width. In the blind holes, of 5mm in diameter and 15mm in length, made in the middle of five lamella, experimentally we placed a live fly larvae in each, that resembled the larvae of House Longhorn Beetle (*Hylotrupes bajulus*), monitoring the eliminating effects of the heating treatment to the larvae. We used a heat camera to check the temperature of the lamellas prior to the treatment and after the 15 minutes long irradiation.

Depending on the irradiated material thickness, the degree of heating up decreased and along the length of the lamella there were two focal points observed. 10 seconds after the treatment, we measured degrees between 35,4 - 111 °C in the edges, and 36,3 - 69 °C in the middle of the impactors. During penetration time we observed thermal degradation of the wood touching directly the applicator, what suggests that the temperature of the wood locally may have exceeded 200 °C during the treatment.

It proves the efficiency of the treatment eliminating the larvae, that during the time of our experiment, the larvae placed in the wood up to 75 + 10 mm material thickness - that is in the 4 lamellas closest to the applicator - all larvae were destroyed. In case the material thickness exceeded 100mms, the applied treatment didn't prove to be efficient, leaving the larva alive in the 5th lamella.

### INTRODUCTION

Microwave treatment is an effective method in eliminating damages caused by wood boring insects. Both free and bound water found in wood can be heated up using microwave radiation. Water molecules start moving because of microwave radiation and this generates frictional heat and causes the wood to heat up. The degree of heating up depends on the dielectric properties of the wood.

Xylophagous insects can be killed at any developmental stage (egg, larvae, pupae, adult) between 45 °C and 65 °C. The success of the treatment depends on the duration of radiation and from the degree of heating up (Makovíny, I., and partners, 2012) The moisture content, the dimensions of the treated wood and the power of radiation all influence the degree of heating up. (Chidichimo, G. and partners, 2018). In their studies Makovíny, I. partners (2012) using 1 W/cm<sup>2</sup> heating power to heat up wood to 50 °C it took 34 minutes to eliminate the house beetle larvae. In the studies of Andreuccetti, D. and partners (1995) the letalis temperature necessary to kill the house larvae was 53,5 °C. Patrascu, M. and partners (2018) treated linden (*Tilia x europeae*) wood with a generator of 1,2 kW at a frequency of 2,45 GHz After heating to 52–54 °C 100% of the house larvae died.

## MATERIALS AND METHODS

During our measurements we examined the efficiency of the MWG-1000-A-1 type microwave equipment for different type and dimension of wood. The radiated power of the 2,45 GHz frequency microwave magnetron generator is 980W. The main parts of this equipment are the mains and connection cables, the timable remote control unit, the generator and the pyramid shaped radiator.

Firstly we conducted our measurements on pine (*Pinus sylvestris*) lamella in which we placed insect larvae. Since we did not have laboratory-housed house beetle larvae we placed common fly (*Musca domestica*) larvae into the holes drilled in the wood.

Our model was made of 6 pieces of planed pine sapling slats. The lamella's individual dimensions were 250 mm × 50 mm × 25 mm. We conditioned the wood for 4 weeks at 20 °C and 65% ± 3% relative humidity. In the middle of 5 pieces of lamella we made blind holes of 20mm deep and 5mm diameter.

In the holes we placed a fly larva in each and we placed the wood on top of each other. We numbered the lamellas from no 1 to no 6, no 1 being the lowest and no 6 the top one. In wood no 6 we did not drill a hole, but we used it to close the model. The average density of the lamellas were 0,76 g/cm<sup>3</sup>. The weight of the fly larvae was 0,77 g/10 pieces.

The treatment lasted for 15 minutes. 20 seconds after switching off the equipment we checked the wood temperature using a Fluke TIR3 type infra camera. (Figure 1).

The measurements were tabulated (Table 1).

The measurements were performed in the NRRC FMK Laboratory of University of West Hungary.

### Equations

The temperature difference ( $\Delta t$ ) was calculated from the average of the temperatures measured at the edges (a) (b) and the centre (c) of the lamella.

$$|\Delta t| = |[(a + b) / 2] - c| \quad (1)$$

## RESULTS AND DISCUSSION

We experienced the most intense heating up in the edges of lamella no 1, that was placed on the radiator of the generator. At the edges of this lamella we measured 111 °C while in the centre it was 69 °C. In the centre of lamella no 5 we measured 41,4 °C. At the edges of piece no 6, that was placed the furthest from the antenna, the temperature was 35,4 °C and 36,9 °C while in the centre it was 36,3 °C. In the edges of lamellas no 1, 2, 3 and 4 the wood warmed up much more intensively than near the holes. The temperature difference decreased continuously moving away from the antenna. In the centre of lamellas no 5 and 6 the average temperature was 0,3 °C higher in the edges. The values measured in the edges and in the middle of the wood showed a decreasing difference moving away from the generator. In the 20 second between the treatment and the measurement the wood slightly cooled down, so during the treatment the temperature of the wood was higher than the measured values. At the edges of lamella no 1, 25 mm away from the radiating antenna the average temperature was 111 °C, while in the middle of the lamella it was 69 °C. The temperature difference therefore was 42 °C. Measuring from lamella no 5,

when counting from the 5th lamella, so in case of wood being placed further than 100mm from the nozzle, the difference between the average temperature measured at the edges and the centre ( $\Delta t$ ) is already a value below 1 °C. The temperature difference ( $\Delta t$ ) measured between the edges and the centre of the lamellas continuously decreased while moving away from the nozzle. Modification and discolouration were also observed on the plate of lamella no 1 where it directly touched the nozzle, and during the treatment the temperature presumably exceeded 200 °C. The larvae died in all for lamellas except for lamella no 5. (Figure 2). The larva placed in lamella no 4 was 80 mm away from the nozzle

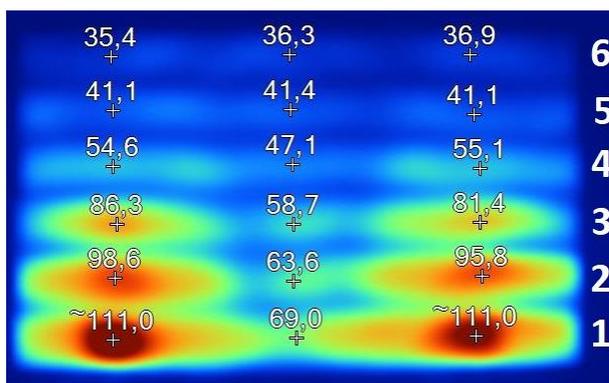


Figure 1: Thermal imaging of lamellas



Figure 2: Lamellas after treatment

Table 1: Results of test

Lamella's serial number	Avarage temperature on the edges [°C]	Avarage temp in the centre [°C]	Temp difference ( $\Delta t$ ) [°C]	Condition of the larvae
1	111,0	69,0	42	died
2	97,2	63,6	33,6	died
3	83,9	58,7	25,2	died
4	54,9	47,1	7,8	died
5	41,1	41,4	0,3	stayed alive
6	36,2	36,3	0,1	-

## CONCLUSIONS

After a 15minutes treatment with the MWG-1000 A type microwave generator all fly larvae died that were placed in the holes drilled in the lamellas except the one in lamella no 5, thus those that were 80 or less mm away from the nozzle. The temperature of the wood was taken 20 seconds after the treatment.

At this moment the temperature measured next to the hole drilled in lamella no 4 was 47,1 °C. In case of lamellas no 1, 2, 3 and 4 up to 100mm away from the antenna, the heating in the edges was more significant than at the centre. The difference between the temperature values measured at the edges and at the centre continuously decreased as in the centre of the lamellas after this the temperature value difference was already below 1 °C. So in addition to the distance from the radiating antenna, the death of the larvae is also determined by their location within the lamellas.

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## Wood response investigation of four selected non-native woody plants to various mechanical injuries

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**Keywords:** Non-native, Woody plants, Mechanical wounds, Response

### ABSTRACT

Humans are an important factor in appearance of non-native plants in natural ecosystems. Because of different specific purposes we planted and spread some species that eventually became invasive. One example is the planting of non-native wood species in areas devastated and damaged by windbreaks, ice breaks and snowstorms, where native species are less successful than pioneer species. When an alien species anchors itself in a suitable environment, it can begin to displace native indigenous species spreading intensely without natural enemies. In Slovenia, non-native species represent around 1 % of the total wood stock, where the largest share black locust (*Robinia pseudoacacia*) has 0,6 %, and in some places appears as invasive. Currently, the most problematic non-native wood species in Slovenia are the black locust and the tree of heaven (*Ailanthus altissima*). Monitoring of black locust, tree of heaven, and other two non-native species, namely Thunberg's barberry (*Berberis thunbergii*) and cherry laurel (*Prunus laurocerasus*), is carried out in Landscape Park in the City of Ljubljana and in Biotechnical faculty test field to assess their response and compartmentalization ability after mechanical damage. In the course of the implementation of four different methods of mechanical damage for control of wooden plants response (cutting, girdling, double girdling and 9/10 ring of girdling trunk), we widen the knowledge on the response of wood plant after injury and on processes that follow chronologically during their growth. The development of a valid method may determine the success in controlling and limiting invasive alien species in our environment and forests.

### INTRODUCTION

Slovenia is characterized by a very well-preserved nature with great biodiversity, which is spread over a varied relief and many habitat types. With the Nature Conservation Act (ZON-E RS, nu. 82/20), we have a basis for the comprehensive conservation of biodiversity and the protection of natural values as heritage. Indeed, we have about 10 % of the territory in protected areas, and almost 38 % is protected under Natura 2000 (Arso..., 2020). It is the responsibility of the state as well as citizens to protect and preserve plant and animal species and their habitats. A concern that is now being highlighted regards the alien species that have been brought to new places ever since man travelled and traded around the world. Most of the species introduced in the new environment do not cause problems, they do not settle down and even less of them become invasive. Rule ten is evident from various sources, its meaning that about 10 % of all non-native species settle in the new area, and 10 % of these become invasive over time, so about 1% of the original number (Williamson and Brown, 1986; Jogan, 2000; Pyšek et al., 2009). However, species that become invasive are one of the most important causes of biodiversity loss and ecosystem instability, as pointed out by the Secretariat of the Berne Convention and the Conference of the Parties to the Convention on Biological Diversity (Act on Ratification of the Convention, 1996). Invasive species combined with current climate change conditions expose indigenous species and their ecosystems to an even greater risk to survival.

#### ***Native and non-native to invasive species***

The native species is defined in the Convention on Biological Diversity as a species, subspecies or lower taxon that lives or occasionally occurs in the area of its usual past and present distribution. This range could be reached by the species on its own, by walking, flying, water, wind, or otherwise (Veenvliet et al., 2009). Alien species are species that are introduced to areas outside their natural range. In this area, however, they

can achieve successful spread without direct or indirect human influence. Thus, with the development and use of means of transport around the world, man enables the transfer and then settlement of plant or animal species through natural barriers, such as oceans, high mountains, etc. into environments where they had never lived before. In the Slovenian legislation of the Nature Conservation Act, the definition for an alien species is: "An alien non-native plant species is one that has been inhabited by humans and was not present on the territory of Slovenia before settlement" (Veenvliet et al., 2009). Invasive species are non-native plant or animal species that spread in natural habitats outside their natural area of growth. In the new area, however, they cause major changes in the composition, structure and function of the habitat, thus either endangering human health, the economy and even indigenous diversity. According to the definition of the Convention on Biological Diversity, an invasive species is one that has stabilized in a new environment and threatens ecosystems, habitats or native species by spreading (Torkar, 2012). These are also species that cause changes in the environment, endanger human health, the economy and indigenous diversity.

Many non-native species do not survive in the new environment in principle, others gradually become domesticated, and some may reproduce and spread more successfully, especially if they no longer have a natural enemy. Thus, in this way, they significantly change the species composition or even the functioning of the ecosystem. Due to such rapid spread and damage, such alien species become invasive as they reduce, hinder, and displace the number of native species. Among the non-native and invasive species, are also reported transitional non-native species that thrive in a certain area and occasionally reproduce, but the population is not replaced by itself, but is linked to repeated introduction into the environment (Richardson et al., 2000; Jogan, 2012).

Typically, the most common non-native wood species are Sitka spruce (*Picea sitchensis*), Douglas-fir (*Pseudotsuga menziesii*), grand fir (*Abies grandis*), eucalyptus (*Eucalyptus* sp.), black locust (*Robinia pseudoacacia*) and others. Their economic benefit and use in the timber industry quickly proved to be advantageous; e.g. eucalyptus in Portugal and Australia, sitka in Scotland (Hasenauer, et al., 2017). In Europe, there are more than 150 non-native wood species found in forests and even more in parks and arboretums. Despite the large number of non-native species, their share per area in the entire European forest is only 4 %. At the same time, there are regional differences, where this share is also less than 1 % in the Balkan countries or significantly large in Ireland and Scotland, which is 60 % (Brus, et al., 2016b). Non-native wood species are not only interesting for primary industry and construction (Gorišek et al., 2018; Gornik Bučar, et al., 2019; Merhar, et al., 2020), but also represent a great growth potential in devastated and affected areas due to the consequences of climate change (Nicolescu, et al., 2020). Douglas-fir, black locust, tree of heaven (*Ailanthus altissima*), ponderosa pine (*Pinus ponderosa*) and red oak (*Quercus rubra*), which are more adaptable, adapt well to these changes (Hasenauer et al., 2017). Although the restoration of the affected forest must be based on native tree species, we have no real guarantee that these species will be successful in the long run, so non-native wood species may in some cases represent a solution in forest regeneration (Brus, 2019). According to data from 2012, non-native wood species represent 0.99 % of the total forest mass in Slovenia, where *Robinia pseudoacacia* predominates with a share of 0.6%, followed by *Pinus strobus* 0.18 %, *Pseudotsuga menziesii* 0.05 % and *Quercus rubra* 0, 03 % (Kutnar and Pisek, 2013). At the same time, we must emphasize the possibility of ecological risk, where new tree species can also have a negative impact on nature, even with the loss of biodiversity (Hasenauer et al., 2017). Tree of heaven (*Ailanthus altissima*) is an example of a species that was first used for afforestation of Kras area (Karst) in Slovenia, but is now spreading invasively into forests and has been added to the list of Regulation 1143/2014 on preventing and controlling the introduction and spread of invasive alien plants. In Slovenia, the natural regeneration of our forests is based, where we have more than 70 indigenous tree species that can be found in more or less specific habitats. The use of non-native tree species for plantations in Slovenia is not common, but it is becoming more frequent (Hasenauer et al., 2017). The example of the Douglas-fir is one of the rare non-native species that has been cultivated in our country for quite some time. It proved to be a very hardy species during ice-storms and subsequent droughts, so it has great potential to be grown on a larger scale. In addition to the already represented non-native species such as black locust, white pine (*Pinus strobus*), douglas-fir and red oak, it is necessary to check the potential of other non-native species. Such findings require decades of testing and knowledge of examples of good practice from European countries. In this way, it is possible to set guidelines for the future of forest management, where it is necessary to comply with legislation and regulations for Slovenia; whereby nature protection is extremely emphasized (Brus, 2014).

In Slovenia, we are not yet dealing with non-native invasive species systematically enough. Recently, the issue of invasive species has been intensively addressed by many projects such as: Life Artemis, campaign “The Gloves Up”, bioportal.si, the Applause project, etc.

### ***Negative impacts of invasive plants***

Invasive species have been one of the main global environmental problems in recent decades due to their negative impact on biodiversity, economy and human and animal health (Vila et al., 2011). Damage caused by invasive species together with the cost of control measures is estimated at 12 billions euros per year on the basis of documented EU information (Regulation 1143/1014; Audit Report, 2019). Similar estimates of the costs of non-native species for the Republic of Slovenia have not yet been made and it seems difficult to determine. In principle, non-native tree species began to be introduced into European forests about 250 years ago. It was a time of rapid technological development and the industry increased its demand for raw materials. A factor without which non-native species would not exist is their introduction into a new area with the help of man. It is man who intentionally or unintentionally helps an organism through a geographical barrier that has so far remained within the area of natural distribution or natural range (Jogan and Kos, 2012). Most of the alien trees introduced at that time are still growing today, as they normally integrated into the new environment and built new stands or even spread across all boundaries, at the same time many of them also perished soon after introduction. After that, they were not talked about for a few decades, but nowadays they are becoming interesting and worrying again due to their high invasiveness. The key to this is the rapid and intense climate change, but we do not yet know what consequences it will have on our forests. There is a possibility that our indigenous tree species will not be able to adapt to new climate change in time. This raises the key question of how recently introduced, forgotten alien invasive tree species can be used for technical purposes and wood processing (Gorišek et al., 2018; Merela et al., 2018; Merhar et al., 2020).

In Austria, the management of invasive species is exemplary. They regularly monitor the emergence of new species in the Austrian flora. Estimates of black locust management orientations in habitats bordering sensitive areas are given (Hulme et al., 2008). A catalog of invasive plant species was published in the Czech Republic in 2002 (Pyšek et al., 2009). At the level of the European Union, a number of projects and initiatives are emerging providing key information on invasive species, examples of good practice, pooling of knowledge and research. At regional level, the NOBANIS network (European Network on Invasive Alien Species) operates, which brings together the countries of Central Europe and represents a source of information for the transmission of effective methods and measures to prevent input. In addition, ongoing projects ALARM (research project on invasive entry routes and habitat risks) and DAISE (where a list of invasive species in Europe and a list of experts most involved in this field have been developed) are well known. Since 2012, the EASIN project (European Aliens Species Information Network), which represents the Alien Species Information Network, has been operating, with the aim of increasing access to data and information on non-native species in Europe, where the Slovenian Life Artemis project also documents its findings. Problematic Invasive Alien Plants (IAPS) are also main topic of the APPLAUSE - Alien Plant Species from harmful to useful with citizens' led activities project. APPLAUSE addresses unsolved questions regarding invasive alien species in terms of the zero waste approach and circular economy. Among other invasive plants also 17 woody plants are examined as potential for wood products (Applause, 2017).

Many researchers (Haisey, 1997; Barry et al., 2001; Roženberger et al., 2017) address the issue of ways to take action against the spread of invasive plants. Chemical control is not allowed in Slovenia (EU Regulation, 2014) as it has a negative impact on the remaining animal and plant population in the area of treatment of invasive species. The only effective and safe way is mechanical removal or suppression by mechanical damage. As a way of optimal control, ringing of tree trunks is highlighted (De Schepper and Steppe, 2013; Merceron et al., 2016; Villa et al., 2017). With the latter method the bark, cambium and the last few bumpers are removed with a tool. The injury techniques themselves vary according to trunk diameter (De Schepper and Steppe, 2013; Roženberger et al., 2017). Research shows that most native as well as non-native invasive species are able to regenerate shoots over and over again, making permanent tree control very time consuming, difficult, and often unsuccessful (Redei et al., 2002; Brus et al., 2016a). In this context, monitoring of black locust, tree of heaven, and other two non-native species, namely Thunberg's barberry (*Berberis thunbergii*) and cherry laurel (*Prunus laurocerasus*), is carried out in

Landscape Park in the City of Ljubljana and in Biotechnical faculty test field to assess their response and compartmentalization ability after mechanical damage. The aim of the research is to investigate the anatomy of tissues after mechanical injury, where we are interested in the response of selected invasive plants. Invasive alien species are limited and eliminated by various measures, thus preventing their rapid spread. We want to determine the optimal way of mechanical damage to the trunk or branch of a woody plant, which is sufficiently effective, weakens the plant and prevents further spread. At the same time, we are trying to develop a tool for the formation of mechanical damage to the trunk.

## MATERIAL AND METHODS

In our study, we selected four wood species (two tree and two shrub species), namely tree of heaven (*Ailanthus altissima*), black locust (*Robinia pseudoacacia*), Thunberg's barberry (*Berberis thunbergii*) and cherry laurel (*Prunus laurocerasus*) of which we evaluate in detail the specific mechanical damages such as cutting and different types of girdling and cutting. At the same time, we chronologically monitor the response of anatomical features in the wood after injury. To pursue the aim, we applied various research techniques such as light microscopy, electron microscopy, magnetic resonance imaging (MRI) First three species were in a nearby landscape park in Ljubljana, while for the fourth specie, cherry laurel, we have seedlings in pots supplied from the nursery and placed in the test field of the faculty. The total number of all four non-native woody plants is 312, including bush species with several damaged shoots (somewhere 430 wounds on trunks or branches), which contributes to the total number of damaged plants to investigate. To compare the injuries, we prepared a special hand tool (Fig. 1), with which we apply the injury on selected specimens, so that the width and depth of the wound are controlled and thus comparable. Therefore, in one method we damage the plant and monitor their response, then we have three forms of girdling's (Table 1). Based on the specific response of each species to different types of damage, we will determine the success of the selected damage method to control and limit species growth. Anatomical studies will help us to understand the response of selected species xylem, as the ability to compartmentalize damage and decay in trees.



*Figure 1: Prepared tool for comparable damage to selected non-native woody plants*

## RESULTS AND DISCUSSION

Numerous injuries to the trunks and branches were induced in the first week of April 2020, where the events following the injury responses are still being monitored today. In shrub species, the first drying and loss of leaves is observed after 80 days from damage. In the case of tree species, instead, most of the damaged trunks began to dry immediately, thereby leaf fall. Below the injury, however, shoots appeared as early as one week after injury. Research shows that most invasive species are able to regenerate shoots over and over again, which can make permanent tree control very time consuming, difficult, and often unsuccessful (Redei et al., 2002; Brus et al., 2016; Villa et al., 2017). As a successful method of restraint and suppression, 9/10 ringing is implemented in our study, where 1/10 of the phloem on the girdling remains (Table 1). That kind of damage weakens tree or shrub considerably and thus slowly depletes it. Significantly less or no new shoots are also observed.

**Table 1: Tree of heaven plant response to different types of mechanical damage**

Tree of Heaven	Cut off	1 x girdling	9/10 of girdling	2x girdling
At the day of mechanical damage				
80 days after mechanical damage				

When the plant was cut, several new lateral buds appeared after only one week, especially in the tree species like heaven and black locust. In the bush species Thunberg's barberry and cherry laurel there are no new lateral buds on the cut trunks and look like that the cut steam had dried. It should be noted that we did not cut the whole bush but only one stem. The other stems may otherwise be mechanically damaged, as shown in the example of Fig. 2, or also without mechanical damage. In the case of 1x or 2x girdling, most of the trees dried up but at the same time under injury new buds or shoots appeared.



**Figure 2: Thunberg's barberry in Biotechnical faculty test field, three shoots, tree mechanical damage (2x girdling, 9/10 of girdling and 1x girdling with new shoot).**

In shrub species, however, less leafing is observed than in undamaged parts of the plant, and the first leaf fall is observed eighty days after some type of girdling. There are no differences between the Tree of heaven plants in the landscape park and the test field at the faculty. Many selected and damaged invasive trees have dried up and decayed, while the shrubs are still growing.

## CONCLUSIONS

Meloche and Murphy (2006) found that the most effective method is felling and simultaneous application of a systemic herbicide, the felling itself does not prove to be effective, as a larger number of new shoots appear next year. Pulling also proves to be a successful method (Meloche and Murphy, 2006), which must be sufficiently effective and in the case of an alien species detected in time. At the moment, the most problematic invasive wood species in our country are the high tree of heaven and black locust, both of which are still locally present and currently do not cause major problems in forests, but at the same time caution and constant monitoring apply. Implementing different methodology, we are seeking to develop the most effective method to achieve success in controlling and limiting invasive alien species in our environment and forests. Our applied result, which is based on a scientific investigation, will therefore contribute to the identification of a reduction in the population of invasive woody species. The research is still ongoing and we are monitoring chronologically damaged plants, their response to damage and the success of each method of damage.

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## Application of an alternating drying schedule for high-quality oak

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**Keywords:** oak, drying, alternating schedule, high-quality hardwood

### ABSTRACT

The drying of high-quality hardwoods is an energy and time demanding practice. A reduction in drying time and energy input while keeping up with quality requirements by the industry is still a major research topic. To tackle this problem a novel drying program with alternating cycles of active and passive phases was applied and tested on oak boards (*Quercus spp.*) with a thickness of app. 50 mm. During the active phases, the wood is dried according to standard process parameters, in the passive phase heating and irrigation are turned off and air movement through the wood pile is reduced. Three drying experiments with different initial moisture contents were conducted with the novel schedule. The drying regime was controlled and operated by a computer-aided process control by regulating temperature and equilibrium moisture content. Moisture content and moisture movement in the boards were measured through resistive measurements and temperature changes inside the boards were recorded with thermocouples positioned in different depths. After the experiments, the moisture distribution and case hardening were determined for quality control of the drying process. The applied schedule shows promising results for future investigations into the intermittent drying of oak as quality defining aspects were excellent and the drying time was in the range of a standard schedule.

### INTRODUCTION

Drying of wood in a standard convective kiln is still the go to method for most big sawmills. Although, other methods, like vacuum or high-frequency drying, show promising results in small scale experiments, the upscaling to bigger dimensions and use in industry has not happened yet (Bond und Espinoza 2016). Due to its special cell structure and high extractives content, oak (*Quercus spp.*) is one of the most difficult to dry wood species. Optimal drying of high-quality oak wood takes a long time and by that binds a lot of money during the drying process (Câmpean und Lazaraescu 2019). Not only maintenance and use of the drying chamber cost a lot of money, also the principal construction. So, a novel idea to, on the one hand improve drying time, energy demand and quality defining parameters and on the other hand use the existing infrastructure would lead to an overall better usage of raw material and infrastructure. The usage of intermittent drying schedules, where phases of high temperature and low equilibrium moisture content (EMC) and phases of rest switch after specified times is one of these ideas. Research into this topic for wood drying dates back at least to the beginning of the 90s and already for quite some time a commercial product for drying softwoods can be found on the market (Langrish et al. 1992). A similar approach can be found in cyclic drying schedules, where the second phase is also controlled actively, by defining different drying climates the conditions in the chamber are regulated and switched regularly (Milić und Kolin 2008; Langrish 2013). The effectiveness of switching cycles of hard and slow drying for better quality and reduced drying time has been shown by numerous studies on different materials (Rufino Franco und Barbosa de Lima 2016; Kowalski und Pawłowski 2011). Intermittent and oscillating drying schedules were already tested and applied to various wood species, although mainly low diameter wood (27 mm or less). Alternating schedules were tested on softwood as well as on more difficult to dry hardwood like eucalyptus and beech and showed good and reproducible results (Herritsch et al. 2010; Langrish et al. 1992; Phonetip et al. 2019).

In this study we developed a novel intermittent drying regime for big diameter high-quality oak wood and tested its influence on the main quality defining parameters. In total three trials with different initial moisture contents were carried out. We hypothesise that, switching phases of active drying and rest of the

wood lead to good drying qualities and drying times. Promising aspects of optimization of the schedule are also discussed.

## EXPERIMENTAL METHODS

### *Material*

Oak (*Quercus spp.*) with a height of roughly 50 mm, lengths bigger than 2300 mm and widths between 200 mm and 400 mm were used for the trial runs. Before the experiments, the boards were cut to a length of 1800 mm and the end grains were sealed with Wood.Protector® by Liquid Solutions to reduce water evaporation. From the left-over boards, two cross sections of 15 mm length were cut off. One was used to determine the moisture content distribution via the oven-dry method, by cutting it into 30 mm wide pieces. At roughly one third of the width one piece was used to determine moisture distribution over the height. The sample was split with a chisel into 8 pieces with a height of roughly 4 mm each. The other cross-section was air-dried and used to compare colour changes.

A stack with the dimensions 1200 mm x 1800 mm x 800 mm was formed out of the boards, between the layers stacking strips (25 mm x 25 mm) were positioned to ensure air flow.

After the drying program 10 boards were tested to determine quality defining aspects. One third of a board was cut off and three cross-sections of 15 mm were cut off. Moisture distribution and colour change were determined as described above. Case-hardening was determined according to ENV 14464 (ENV 14464:2002). The number of boards used for each experiment and the moisture contents pre drying and post drying can be seen in *Table 4*.

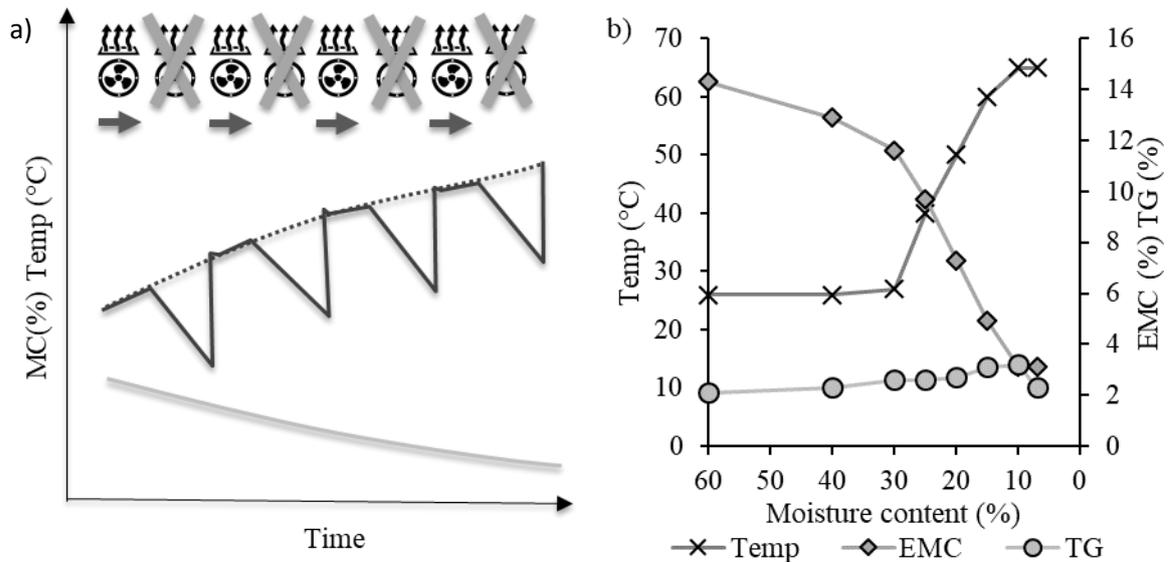
*Table 4 Amount of boards in each trial, mean moisture content of the samples and standard deviation before (pre drying) and after (post drying)*

	<b>Boards in experiment</b>	<b>MC pre drying (%)</b>	<b>Std. deviation</b>	<b>MC post drying (%)</b>	<b>Std. deviaton</b>
Trial 1	27	17,2	1,2	9,4	1,2
Trial 2	25	20,0	3,8	8,0	1,1
Trial 3	26	38,1	5,4	8,5	1,4

### *Drying schedule*

The drying experiments were performed in a laboratory convection dryer operated by a computer-aided process control (MB8000, Mühlböck Trocknungstechnik, Austria) Conditions inside the kiln were controlled via equilibrium moisture content (EMC) and temperature measurements. Ten electrodes for resistive moisture measurement were screwed into seven different logs. In three logs two electrodes were screwed in with different screws (30 mm and 15 mm long) to reach two depths (app. 25 mm and 10 mm) to record moisture differences. Only the electrodes reaching the middle of the boards were used for moisture-controlled regulation of the kiln. Additionally, thermocouples were inserted in the three boards with two electrodes in similar depths to also record the temperature differences over time. Temperatures were logged every minute on external data loggers (PCE-T390). Drying data was recorded automatically every five minutes.

*Figure 10* left shows the theoretical behaviour of the drying schedule. Active phases switch with passive phases every six hours, which leads to the dropping temperature. The active phase is programmed after a conventional schedule (for details see *Figure 10* right) and in the passive phase the chamber is mostly shut down, only the ventilators still run at reduced speed (roughly 25 % of the maximum rotational speed). While other studies are working with active alternating cycles, e.g. different relative moisture contents every other hour, the goal of this study is to use a simple drying schedule where the alternation is provided by letting the wood rest and cool down (Phonetip et al. 2019; Herritsch et al. 2010). Target moisture content at the end of the process was 9 %. After the mean moisture content reached 9 % a conditioning phase was held at 10,6 % EMC for 49 hours.



**Figure 10** a) concept of the intermittent drying schedule with alternating temperature profile and theoretical steady dropping moisture content profile b) drying schedule of the active phases; temperature (Temp), drying gradient (TG) and equilibrium moisture content (EMC) in the chamber at different moisture contents of the wood

## RESULTS AND DISCUSSION

### Comparison of experiments

**Figure 11** shows the main parameters for comparison of the three drying experiments, moisture content (MC), temperature (Temp) and equilibrium moisture content (EMC). To compare the duration of the drying time, the MC curves of trial 1 and trial 2 were aligned with the MC curve of trial 3. Trial 1 and trial 3 take almost the same time for drying the last 7 % of moisture, but trial 2 takes app. 230 hours longer. The temperature curve shows that this deviation is due to a lower drying temperature. The longer drying times can be explained by a slower drying of the material, as the temperature rise is controlled by the loss of moisture content in the boards. Slower drying times can be the result of anatomical features (Hansmann et al. 2002). Deviations due to the intermittent schedule start to get more pronounced after 1100 hours in trial 3 with a maximum deviation at the highest temperature of 65 °C. Due to automatic spraying of water, setting in hard at 1100 h, stronger moisture content deviations can be seen (see **Figure 12** a and b for more details). This also results in a change in the steepness of the moisture content curve, which leads to longer drying times.

In the passive phases the dropping temperature leads to a rise of the EMC, which should have positive effects on the properties of the finished products, as every passive phase can be considered as a conditioning phase for the wood, where the moisture can equilibrate over the cross-section and stress relaxation is happening (La Cruz-Lefevre et al. 2010). In these trials the duration of active and passive phases was the same and constant over the whole drying process. This is quite uncommon compared to other studies, where the passive phases were longer in most cases. These longer passive phases result in more time for the wood to rest and equilibrate the MC over the cross-section (Phonetip et al. 2019). In the first 1200 hours of trial 3 the MC is way above fibre saturation. For oak the standard conditions under these circumstances are a slow, low temperature drying (compare with **Figure 10** b). Temperature and moisture deviations between active and passive phases are also relatively small during this stage. Higher temperatures for a shorter time and long resting phases at ambient temperatures could help reduce the time needed to dry until fibre saturation but also ensure the quality of the dried material. The tendency of oak to show intense discolorations, if it is dried at high temperatures while still having a high moisture content has to be taken into account as well (Câmpean und Lazaraescu 2019).

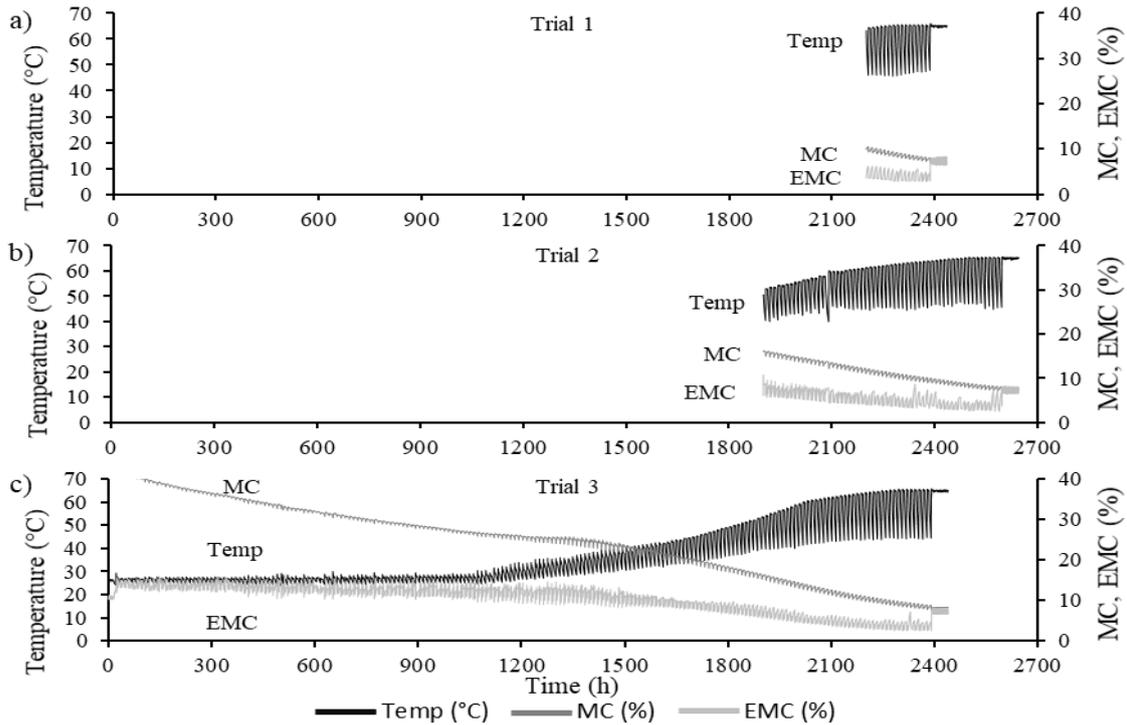


Figure 11 Temperature, moisture content and equilibrium moisture content profile of the 3 experiments. a) trial 1, b) trial 2 and c) trial 3

**Temperature and moisture variations**

The temperature and moisture content variations in 10 mm and 25 mm depths in one board of wood can be seen in Figure 12 a and b. The shown data is from two different times in trial 3. The time it takes to heat up the whole board sums up to roughly 3 hours, so half of the active phase is used to heat up the boards. A possible optimization step would be the reduction of the active time. Temperature and moisture content variations go hand in hand with the active and passive phases of the schedule. The high variation of the moisture content in Figure 12 a in 10 mm depths is the result of water spraying (not shown in the diagram), in 25 mm depths the impact is much smaller. In the later drying stage, the impact of water spraying on the moisture content cannot be seen anymore.

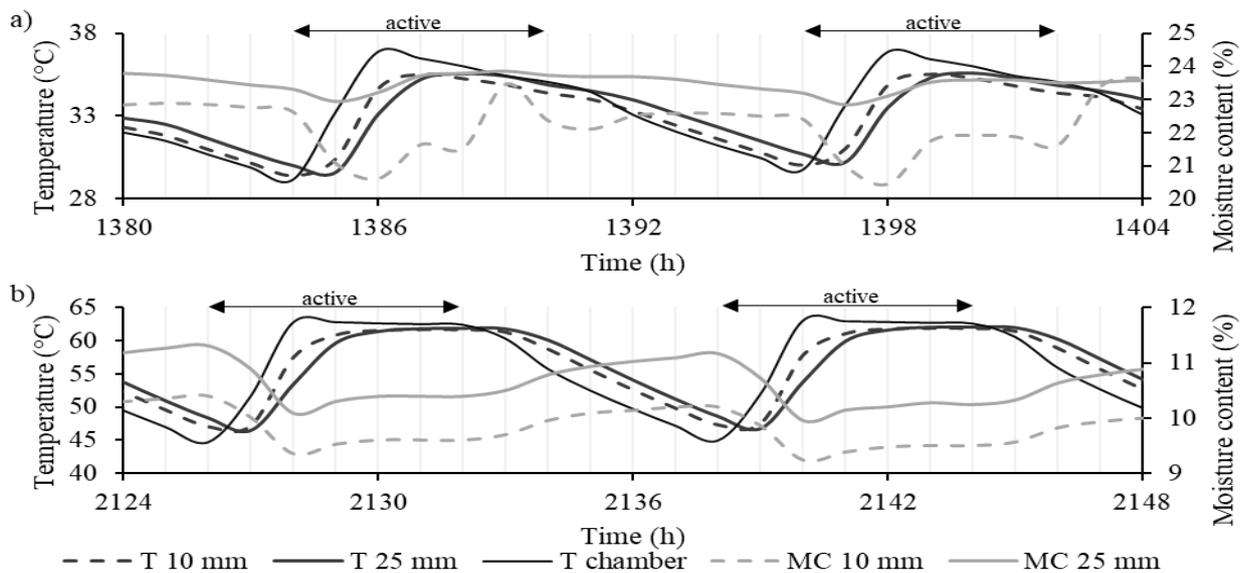
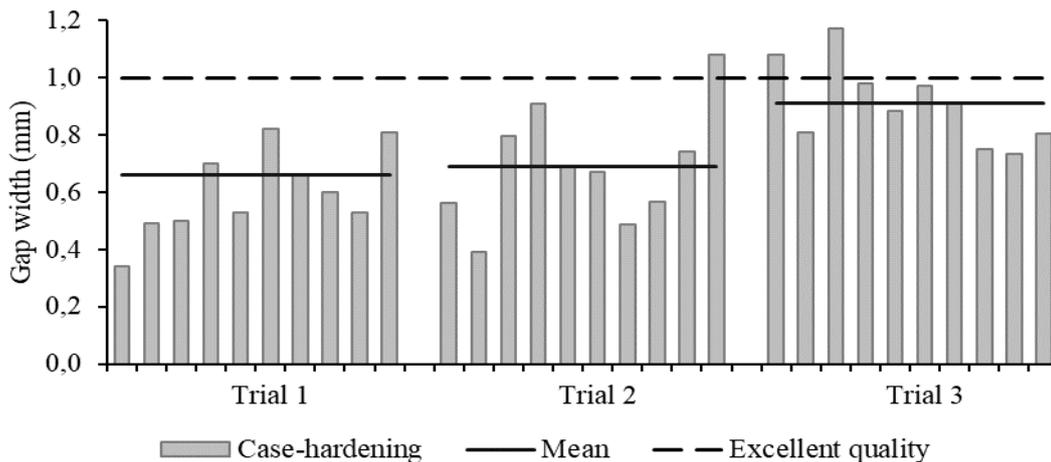


Figure 12 Temperature and moisture content profile inside one board in two depths (10 mm and 25 mm) at two different times during trial 3 a) 1380 - 1404 hours b) 2124 - 2148 hours

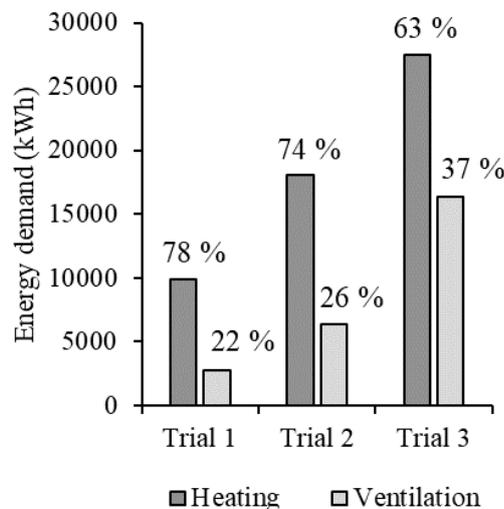
**Quality defining aspects and energy demand**

The quality of the process was tested by measuring the moisture distribution (*Table 4*) before and after the trials and by determining the case hardening (*Figure 13*) of 10 example boards. Except for experiment 3 all samples were of excellent quality concerning the case hardening, mean gap width was app. 0,7 mm. For trial 3 the mean gap width was roughly 0,9 mm, two samples had a gap width of more than 1 mm but overall the quality is still excellent according to ENV 14464:2002. Combining the results from moisture distribution and case hardening we conclude that quality expectations can be reached with the used drying scheme.



**Figure 13 Case-hardening values (mm gap width) of 10 boards per trial run. Indicated as a solid line is the mean value per trial and the value for excellent quality as a dashed line**

Energy demand for the heating and ventilation of the drying chamber can be seen in *Figure 14*. For longer drying times the distribution of the energy between ventilation and heating changes. As long as the MC of wood is above fibre saturations (app. 30 % MC) the drying temperatures are kept below 30 °C to prevent discolorations and stress issues in the boards (Câmpean und Lazaraescu 2019). This leads to a higher energy demand for the ventilation, because almost no heating energy is used while the MC is above 30 %. A potential optimization of energy demand lies in the further reduction of ventilation speed during active and passive phases.



**Figure 14 Energy demand for heating and ventilation in kWh for the 3 trials. Numbers above the bars indicate the allocation of the whole energy consumption in percent to heating or ventilation**

## CONCLUSIONS

A novel alternating drying schedule was developed and tested on high-quality oak wood with three different initial moisture contents. By alternating between active and passive phases, where the drying chamber is shut completely down except for reduced ventilation, it is possible to get good and reproducible moisture distributions and case hardening values. To determine the possible reductions in energy usage comparisons with a continuous schedule are planned. The influence of shorter active phases and higher number of phase switches is part of future investigations.

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## Digital twin of beech plywood based on individual veneer specificities to model their deformation

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**Keywords:** Plywood, Beech, Drying, Numerical simulation, Moisture transfer, Photogrammetry

### ABSTRACT

The main goal of this research program hold by Brugère Company is to model the deformation of plywood panels and 3D shells considering local veneer specificities. The proposed model novelty relies on the impact of the local initial veneer moisture content and the local fibre orientation. The local fibre angle map of each green veneer is measured using laser and tracheid effect (Nyström 2003). The local moisture content will be measured before and after drying using a specific apparatus under development. The plywood global deformation after drying is assessed using the photogrammetry method to build-up a 3D model for its comparison with the Finite Element Model (FEM) digital twin of the same plywood configuration. This paper presents the model structure and the first experimental campaign aiming to validate the FEM and to extract first indications about each parameter influence. The results will be later used to extend models usage to the simulation of beech plywood 3D shells as presented in Figure1.

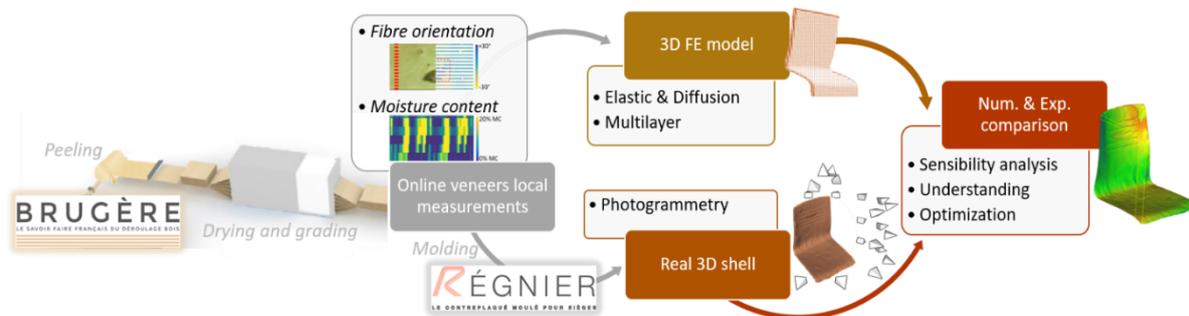


Figure 1: Synoptic diagram of the study and industrial partnership contributions

### INTRODUCTION

Even though plywood shell have undergone the same process with identical parameters, some may feature important differences in geometry once delivered to the customers. This observation was done by Régnier (Part of « Les Manufactures Février » Brugère Compagny being also a part of this group) a French manufacturer specialized in the production of moulded plywood parts. Their production are particularly focused on 3D molded beech plywood shells. A significant part of their production undergoes twist deformations and is therefore discarded. This deformation occurs often when the shells are already delivered to the costumers after few weeks which is particularly detrimental in terms of cost and reputation for the group.

The main goal of this research program is to model the deformation of plywood shells. The study is firstly performed on flat plywood panels to build a method transferable on 3D shells. Thus, according to the practical experience of Régnier Corp, the main parameters responsible for the plywood out of plane deformations are selected to drive the numerical model set up. Indeed, the shape stability depends on a variety of materials, veneer and plywood process parameters. According to operator's knowledge, the first reason for the plywood cupping/wrapping seems to be the inhomogeneous moisture content (MC) among the veneers composing the plywood and within each veneers too as already shown by (Bloqvist 2013) who made the same observations. The second influencing parameter is the variable thicknesses of the faces. Before the gluing, the faces are sanded and it may happen that the sanding operation is not symmetrical between the top and bottom faces. According to the Régnier company operators, the over sanding of one face (compared to the other one) increases considerably the risk of twisting. The third parameter is the grain angles since the wood shrinks differently depending on its grain orientation due to its anisotropic and hygroscopic nature. The influence of the global veneer orientation on the deformation of 3D plywood shells has already been studied by (Sandberg and Ormarsson 2007). Those authors propose a numerical model showing that veneers orientation needs to be controlled to prevent the shell from twisting, especially for the external veneers. (Bloqvist 2013) also concludes that the fibre orientation in veneers and between veneers is one of the key factors for the shape deformation especially for the twisting. Reaction wood too, exhibiting until 10 times swelling-shrinkage coefficients, has been proven to strongly influence the final shape of wood engineered products (Sahlberg, Salme'n and Oscarsson 1997, Shmulsky and Jones 2011) due to its particular microfibril orientation in the S2-layer. But the identification of the tension wood on veneers is up to now difficult to realise online and makes the test complicated. Facing that complex multi-physical problem including moisture content, grain direction, intrinsic parameter of each veneer, a Finite Element Model has been developed aiming to develop a robust plywood panel numerical model able to replicate the 3D-shell behaviour.

## EXPERIMENTAL METHODS

### *Sampling*

According to the literature review and the operators observations, it was decided to focus on the initial moisture content, the thickness and the position of each veneer inside the plywood panel. The Table 1 summarizes the compositions tested for the numerical and experimental approaches of the study; a total of 24 panels were prepared. For each tested composition (veneer thickness, veneer moisture and their relative position within the panel), three plywood panels were made. The deformation of plywood is measured at two moisture content, 10% and 6%. To reach out these moisture contents they are placed on a climatic chamber set at (20°C ; 56% RH) for 10% MC and (45°C ; 35% RH) for 6% MC.

The samples arise from 4 logs from the same beech trunk. Their diameter are under 50 cm, considered of low quality for rotary peeling process, with heart cracks and many knots. The logs were firstly steamed in a water tank during 48 hours at 80 °C and then during 24 hours at 50 °C. The logs were peeled on the LaBoMaP instrumented peeling line into veneers at two different thickness, 1.2mm and 0.8mm. The compression rate used in this study was 10% of the initial thickness. The format after peeling was 570 × 520 × thickness mm to reach a dried final format of approximately 500 × 500 × 1.20 mm. After peeling, only the veneers without singularity (knots, cracks) were kept for the present study not to introduce singularities into the modelization as a first step (later on, after validation of the model, they will be considered too but not in the present study). Some of veneers were placed in a climatic chamber to reach 16%MC (15°C, 80%RH) until stabilization.

5 veneers were glued together with cross grain to compose different plywood configurations. The glue used is PVAc (polyvinyl acetate) spread at 240g/m<sup>2</sup>. Plywood were pressed with an Orma press under a pressure of 0.5 N/mm<sup>2</sup> for about 60 min at ambient temperature. After gluing, the panels were squared at their final dimension of 480 × 480 mm<sup>2</sup>.

(Bloqvist 2014) stated that when the tight sides of both outermost veneers are in the same position it minimises the final plywood deformation. However, this composition is generally not used by manufacturers because of the risk of cracking of the veneer surface. Therefore, for this study, the tight side of both faces are placed in the opposite positions. The rest of the veneers are in the same direction.

**Table 1: Plywood composition**

Combination		Layer 1	Layer 2	Layer 3	Layer 4	Layer 5
A	Thickness	1.2	1.2	1.2	1.2	1.2
	Initial MC (%)	16	9	9	9	9
B	Thickness	1.2	1.2	1.2	1.2	1.2
	Initial MC (%)	9	16	9	9	9
C	Thickness	1.2	1.2	1.2	1.2	1.2
	Initial MC (%)	9	9	16	9	9
D	Thickness	0.8	1.2	1.2	1.2	1.2
	Initial MC (%)	9	9	9	9	9
E	Thickness	0.8	1.2	1.2	1.2	1.2
	Initial MC (%)	16	9	9	9	9
F	Thickness	1.2	0.8	1.2	1.2	1.2
	Initial MC (%)	9	16	9	9	9
G	Thickness	1.2	1.2	0.8	1.2	1.2
	Initial MC (%)	9	9	16	9	9

***Plywood deformation assessment by photogrammetry***

The global deformation after drying is assessed using the photogrammetry method to build-up a 3D model. This technology presents two advantages which have led this choice. The first one is the low cost since it only needs a quite standard digital camera and a minimum of equipment. The second is that it's easy to transport and it is possible to use this system in the partner's production sites. One drawback could be the treatment time which is too long to directly transfer onto an online measuring system.

A set of approximately sixty pictures around one plywood panel were taken using a canon EOS2000D camera equipped with a EF-S 18-55mm f / 3.5-5.6 IS II lens. The photogrammetry uses the technology of the image correlation which consists in the automatic recognition of homologous pixels on a defined surface. The software performs a stereoscopic readout of the scene to determine the relative positions of each point. In this study Autodesk Recap photo is used. The multiplication of the process at a large number of points of view makes it possible to make the position calculations of each pixel more reliable by dividing the error while increasing the extent of the 3D modeling. To ensure an accurate 3D reconstruction , it is important to take enough pictures with homologous pixels at different angles of view. In this study a picture was taken every 20° at three different plunge angle. It is important to respect some rules to ensure the right running of the software. The picture needs to be clear of blur as much as possible on the whole image. Parameters of the camera have to remain constant during the whole series of pictures. The calculation takes about one hour after what few minutes of post treatments are required to remove the background as describer in Figure 2. The numerical 3D model can be exported in many file formats as STL (as in Figure 1).



**Figure 2: 3D model of a plywood panel obtain by photogrammetry method**

The shape of each panel was measured three times. The first one is acquired instantly after the pressure is released and the two others ones were performed respectively at about 6% and 10% MC thanks to the climatic chamber as described previously.

### ***Numerical model setting***

In this study, the numerical model uses the Finite Element Method approach with Abaqus software and the geometrical model automatically computed by a script made with Python. This Script is useful to easily change the model parameters.

### **Wood shrinkage and mechanical parameters**

The displacements are approximated from nodal displacements and depend on the type of elements considered. In this model, hexahedral elements with twenty nodes are used (C3D20RT). The shrinkage is considered as orthotropic linear from the fibre saturation points to 0% MC. According to (CIRAD 1998), the values used are 0.36%/ % in the tangential direction, 0.18%/ % in the radial direction and 0.03%/ % for the longitudinal direction. This method enables to consider the grain local direction angle effect. The stiffness parameters in Table 2 used in the simulations are based on data presented by (Guitard 1987).

**Table 2: The stiffness data used for beech (*l* – longitudinal, *r* – radial and *t* – tangential).**

$E_l$ (MPa)	$E_r$ (MPa)	$E_t$ (MPa)	$\nu_l$	$\nu_l$	$\nu_l$	$G_{lr}$ (MPa)	$G_{lt}$ (MPa)	$G_{rt}$ (MPa)
13900	6000	2000	0.64	0.29	0.26	650	650	1500

The geometry is a single parallelepiped with the external dimension of the plywood. The layers are modelled by setting the local orientation of each elements on the basis on their position in the parallelepiped. The advantages of this method is to make easy the changes in the number of layers and their dimensions. The actual glue joints between veneers are not taken into account because they are assumed to have a significant lower impact than the moisture content and the grain angle (Bloqvist 2013). Moreover, it is difficult to find studies that quantified the impact of glue joint on plywood deformation and water diffusion.

### **Moisture flux inside the wood**

The water transport following equations come from a method presented in (Fortino 2009). Authors used an analogy between heat flux and moisture transfer in the wood. The wood is assumed to follow the second Fick's law for the moisture transfer (Eq.1). The temperature is considered to be constant.

$$\left. \frac{\partial MC}{\partial t} \right|_{\Omega} = \nabla \cdot (D \cdot \nabla MC) \quad (1)$$

where  $MC$  is the wood moisture content,  $D$  the second-order diffusion tensor of moisture transfer and  $\Omega$  the exchanging volume considered. This form of Fick's equation can be used when the density is constant. The beech wood is considered as a homogeneous specie since the difference between earlywood and latewood is moderate. So the density is taken at 710 kg/ m<sup>3</sup> (CIRAD 1998). The diffusion coefficients are computed from (Sjodin 2006) for the longitudinal direction  $D_L$  and for the transversal direction  $D_T$ . The transfer in the radial direction and transversal direction are assumed to be identical, so  $D_T = D_R$ . The diffusion coefficient dependency with the moisture content in radial and tangential direction is defined in (Eq. 2):

$$D_R(MC) = D_T(MC) = 8.64 \times 10^7 e^{4MC} \quad (2)$$

where  $D$  is expressed in m<sup>2</sup>h<sup>-1</sup>. The (Eq.1) is mathematically analogous with the heat transfer equation (Eq. 3) when considering  $C_T = 1$  and  $\lambda = \rho \cdot D$ .

$$\rho C_T \left. \frac{\partial T}{\partial t} \right|_{\Omega} = \nabla \cdot (\lambda \cdot \nabla T) \quad (3)$$

where  $\rho$  is the density,  $C_T$  the specific heat,  $T$  the temperature of the wood and  $\lambda$  the second-order thermal conductivity tensor.

### **Moisture flux at the wood surface**

The moisture transfer between wood and air is expressed in the Eq. 4 as presented in (Avramidis and Siau 1987).

$$\frac{q_n}{\rho} = S_u(MC_{air} - MC_{surf}) \quad (4)$$

where  $q_n$  represents the moisture flow across the boundary,  $MC_{surf}$  is the moisture content of the wood surface and  $MC_{air}$  the equilibrium moisture content of wood corresponding to the air humidity which. Additionally, the Eq. 5 improves the Eq. 4 by taking into account the coefficient of surface emission  $S_u$  dependency with the moisture content at the surface (Hanhijärvi 1995).

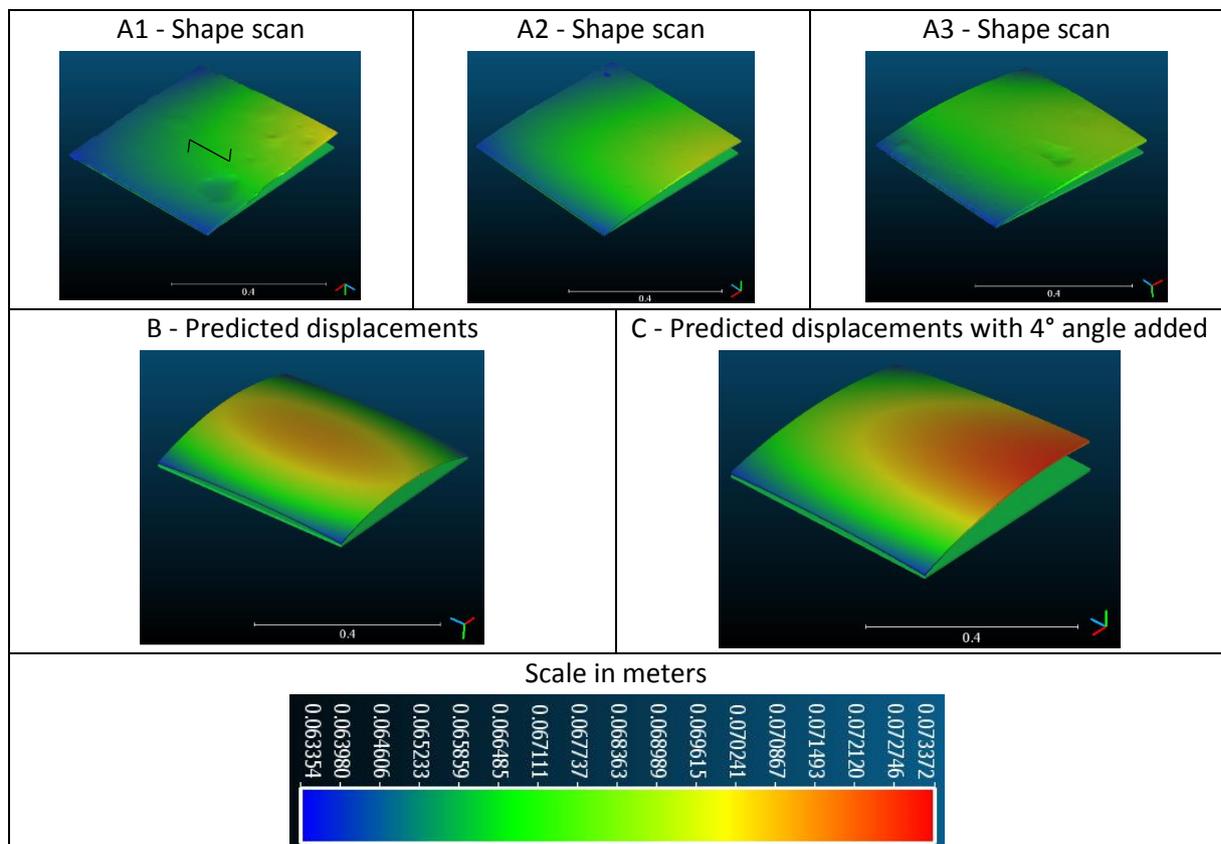
$$S_u = 3.2 \times 10^{-8} e^{4MC} \quad (5)$$

### ***Numerical and experimental 3D models comparison***

In order to interpret the results of the three-dimensional scans and to assess the prediction ability of the numerical model, the CloudCompare open source software is used (<https://www.danielgm.net/cc/>). It is a 3D point cloud editing and processing software design to compute the distance between one three-dimensional object and a reference surface.

## **RESULTS AND DISCUSSION**

Only a part of both the experimental and numerical approaches are presented to analyse the effect of the key factor identified (Tab. 1). The results presented focus on the shape of the plywood for a moisture content of 6%. Fig. 3 presents measurements and deformation for the combination (A) where the thickness are equals for all layers and the moisture content is higher for one outer layer.



**Figure 3: 3D views of plywood panel for combination A: Real shape of the three repetitions (A1-A2-A3), shape predicted by the numerical model (B), and shape predicted by the numerical model with tilting angle (C)**

The combination (B) gives similar shapes with lower amplitudes. This modality helps to understand the MC influence. For the thickness effect (Fig. 4), the combination (D) is presented where the moisture are equal for all layers and a thickness of 0.08 mm for one outer layer. Finally, in the Fig. 5, the combination (E) is proposed, where the outer layer is 0.8 mm thick and at 16% of moisture content. For each following feature (Fig. 3 to 5), 3 kind of results are presented: A<sub>i</sub> are the shape of the three measured panels with the same configuration (repetitions); B is the results of the Abaqus simulation of the respective model, and C<sub>i</sub> are the simulation with some fibre angle corrections (2 and 4 degrees tilting). The plywood are all oriented on the same way. The longitudinal direction are oriented like the indication on (A1) of the (Fig. 3).

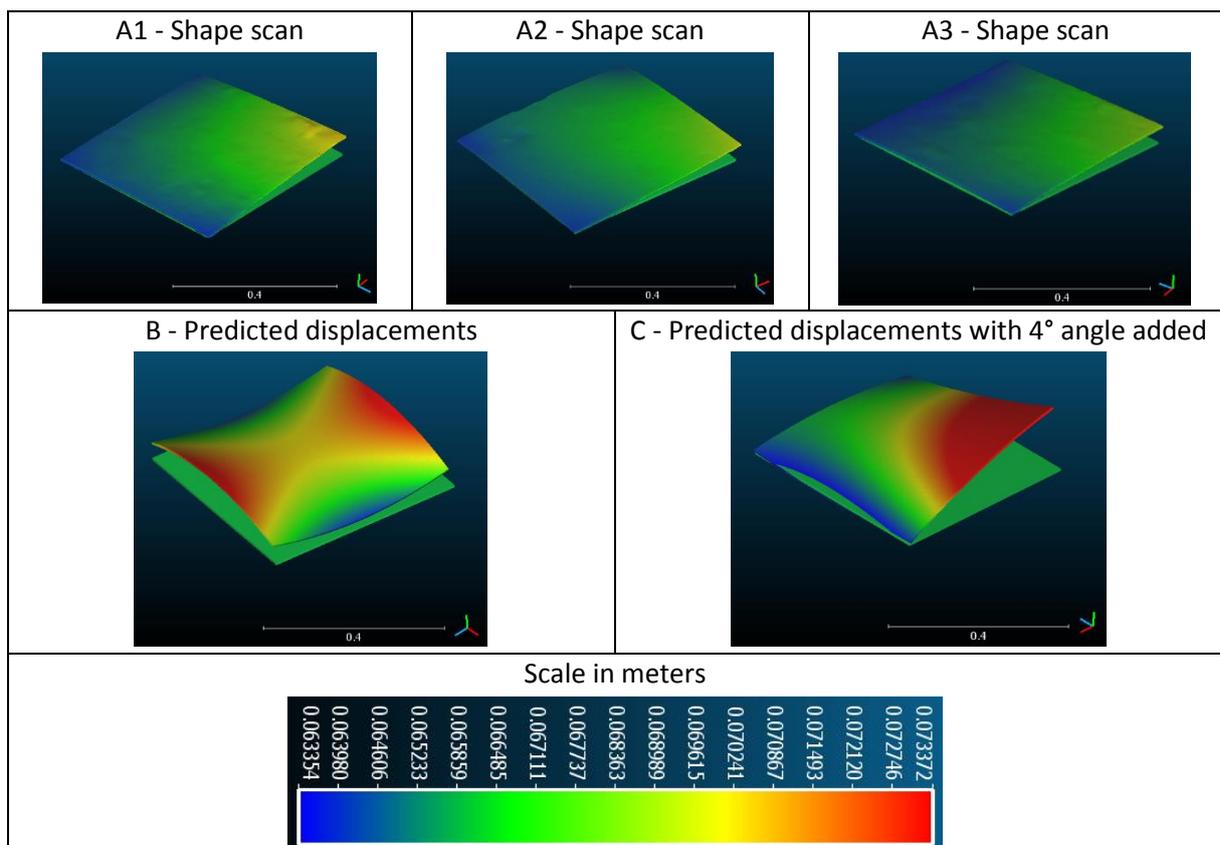
**Moisture content variation.**

As shown in Fig. 3 the behaviour of the model and tests are significantly different. The shape in the three repetitions are similar and the maximum deflection difference is of about 45 mm. The strongest hypothesis to explain the inaccuracy was the issue of mastering the global grain direction when the veneers were glued. In fact, an error of few degrees can occur when the plywood are pressed.

Accordingly, the shape C are with 4° added to the global angle of the first two veneers [88°,2°,90°,0°,90°]. The addition of such tilting angle tends to increase the model correspondence with the experimental behaviour.

**Thickness variation of the face**

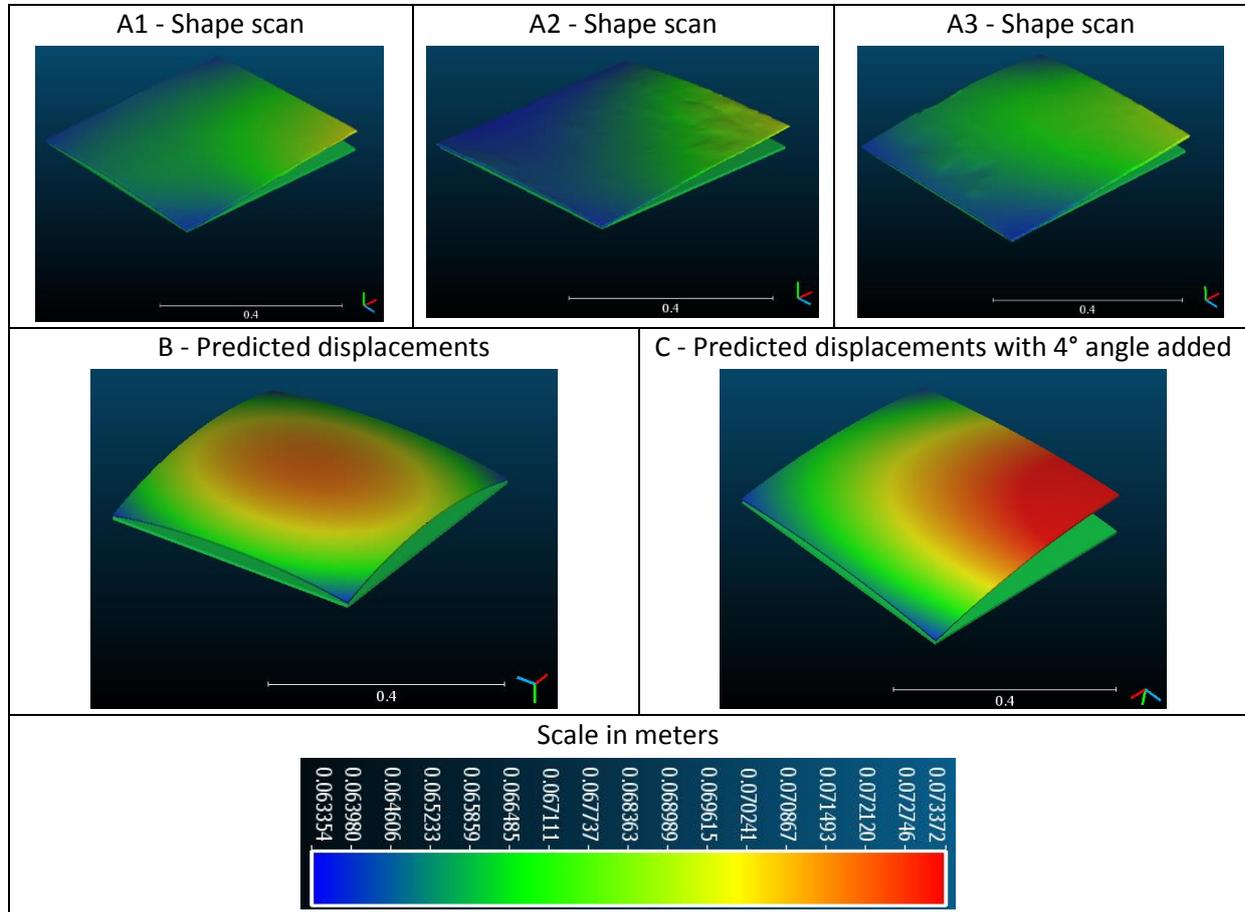
The Fig. 3 represents similar results than the Fig. 2 but focussing on the thickness effect analysis. The behaviour of the model and tests are significantly different as well. But the shape predicted with the model in B is highly different. Because of the same reason, a tilting angle has been added to the numerical model. The shape also seems to tend toward the shape of the test.



**Figure 4:** 3D views of plywood panel for combination D: Real shape of the three repetitions (A1-A2-A3), shape predicted by the numerical model (B), and shape predicted by the numerical model with tilting angle (C)

**Combination of moisture and thickness variation**

The observations made in Fig. 2 and Fig. 3 are the same than the one in Fig. 4. The deformations calculated by the numerical model are significantly different compared to the experimental tests. But once again with addition of a tilting angle on numerical test, the numerical shape tends to behave similarly than the real tests.



**Figure 5: 3D views of plywood panel for combination E: Real shape of the three repetitions (A1-A2-A3), shape predicted by the numerical model (B), and shape predicted by the numerical model with tilting angle (C)**

**CONCLUSIONS**

The results of this study show the leading role of the fibre orientation on plywood deformations. Therefore, it is really important to consider the global fibre orientation to propose a numerical model able to mimic the plywood behaviour. In this study it is not possible to find the exact value of the angle for each layer of plywood even if the local grain deviation of each veneer was recorded just after peeling. Indeed, during pressing, veneer can slightly slide and/or rotate responsible for the apparent inaccuracy between the numerical model and the experimental measured shapes. Finally, the numerical model will be calibrated using 3 ply veneers which makes possible the scan of the local grain angle on both critical surfaces after gluing and pressing.

**ACKNOWLEDGEMENT**

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## Functionalisation of beech wood using magnetic nanoparticles

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**Keywords:** *Fagus Sylvatica*, magnetite, nanoparticles, magnetic, functionalization

### ABSTRACT

New functions of wood may lead to innovative products and the usability of bio-based materials. This study deals with the analysis of magnetic functionalization of beech and poplar wood.

Samples of size 20 x 5 x 10 mm were impregnated with Fe<sub>3</sub>O<sub>4</sub> nanoparticles synthesized with either an oleic acid coating or uncoated. The oleic acid coated particles were suspended in linseed oil as impregnation medium. The uncoated particles were dissolved in a tannin-hexamine solution for infiltration and fixation. Different impregnation parameters (e.g. alternation of vacuum or vacuum-pressure cycles and number of cycles) were investigated. To evaluate efficacy of the procedure the wood samples were placed onto a magnet after curing and assessments on e.g. maximum tilt of the magnet at which the sample was still hold in place were made. Other investigations dealt with the penetration depth of the magnetite and the carrier solution as well as the distribution within. Results show that a magnetite NP content of 5wt% in linseed oil results in pleasing magnetic properties while not altering the optical appearance of the impregnated beechwood specimen in a negative way. The so obtained product could be used for different applications (e.g. bio-based giveaways and office products).

### INTRODUCTION

Modifying the properties of wood to make it suitable for other applications has been done a lot in the past few years. For example removing (Zhu et al. 2016) or modifying (Li et al. 2017) the lignin and stabilizing the remaining structure in PMMA resulted in almost transparent wood, while impregnation with glycerol or other fluids with matching refractive index produced translucent samples. Also conductive wood surfaces were produced by coating with silver nanoparticles (Gao et al. 2016) or the whole specimen was rendered conductive to function as electromagnetic interference shielding (Gan et al. 2020).

Some studies on rendering wood magnetic have been conducted. There are generally two different ways to introduce magnetite nanoparticles into the wood. (i) in situ particle formation, (ii) ex situ particle production followed by impregnation. The first route involves impregnation with an iron salt solution followed by ammonia solution treatment to form the particles (Lou et al. 2018; Gan et al. 2015). The treatment with aqueous ammonia solution might interfere with some wood-species and therefore is not suitable for all of them. For the second route, which is also featured in this study, the particles are synthesised externally and afterwards dispersed in a suitable impregnation solution which is then impregnated into the wood (Gan et al. 2017).

Beech and poplar are two wood species that are suited very much for impregnation as their treatability is 1 according to EN 350:2017. Linseed oil impregnation of wood has been used for centuries to make the wood more durable and resistant to wear and tear. Tannin-based wood impregnation was studied in the past to be a suitable alternative to metal based wood preservative systems (Thevenon et al. 2009; Tondi et al. 2012; Sommerauer et al. 2019). Aim of this study was to evaluate the suitability of oleic acid coated and uncoated magnetite nanoparticle for the impregnation of beech and poplar wood using either an oil- or water-based system.

## EXPERIMENTAL METHODS

### ***Materials***

Chemicals used for the synthesis of the magnetite nanoparticles were FeCl<sub>3</sub> hexahydrate (Roth, Germany), FeSO<sub>4</sub> heptahydrate (Roth, Germany), NH<sub>4</sub>OH 25% (Merck, Germany), Oleic Acid (Merck, Germany). Linseed oil for impregnation was provided by Adler (Austria). For the tannin-based impregnation system condensed tannin Weibul AQ (Tanac S.A., Brazil), Hexamethylenetetramine 33% (Alfa Aesar, USA) and NaOH (Roth, Germany) were used.

Beechwood specimen were cut to dimension of 20x5x10mm, poplar plywood was cut to 30x10x4mm and conditioned for an appropriate time in standardized climate (20°C and 65% r.h.). Their weight was recorded using an analytical balance with an accuracy of 0.1mg.

### ***Preparation of magnetite and impregnation solutions***

To produce hydrophobic magnetite, a procedure reported by (Faridi-Majidi et al. 2006) has been used. In short, 9.72g FeCl<sub>3</sub>·6H<sub>2</sub>O and 6.68g FeSO<sub>4</sub>·7H<sub>2</sub>O were dissolved in 80ml water and heated to 90°C, afterwards 20ml NH<sub>4</sub>OH and 2g oleic acid were added. The mixture was stirred vigorously for 3h and afterwards the magnetite was collected using a strong magnet and washed several times with water and isopropyl alcohol. Those particles were used for the linseed oil-based impregnation solution. For the water-soluble particles, the steps were the same as above, without adding the oleic acid, leaving them uncoated. These were later on used for the tannin-based system.

Tannin solution was prepared as described previously (Tondi et al. 2012) without boric acid by dissolving 15% tannin in water followed by the adjustment of the pH to about 9. Afterwards 6% of aqueous hexamine solution (33%) was added based on the tannin weight.

Concentration of magnetite in the impregnation solution was chosen as 2.5%, 5% and 10%. The oil-based solution was stable for several weeks in a cold, dark storage while the tannin-based solution had to be prepared fresh daily and used immediately after preparation. As tannin tends to form complexes with different metals and metal oxides and therefore lead to precipitation or of the magnetite or gelation of the impregnation solution (Slabbert 1992; Çakar et al. 2016; Meikleham et al. 1994).

### ***Impregnation procedure***

Two different approaches were used for the impregnation of the wood samples. The first one was only based on vacuum, while the second one consisted of alternating vacuum/pressure cycles.

For the vacuum-based setup the samples were submerged in the impregnation solution in a beaker so that all of them were sufficiently covered. The beaker was transferred into a desiccator and a vacuum of 50mbar was applied for 15min allowing the air trapped inside the specimen to be evacuated. Afterwards the vacuum was released, allowing air to flow back into the desiccator for 5min. These cycles were repeated either once (Vac1), twice (Vac 2) or thrice (Vac 3), to evaluate differences in loading and magnetic properties.

For the vacuum/pressure test runs, the samples were prepared as before, but not placed in a desiccator, but in a pressure vessel allowing both, vacuum and high pressure. As a first step, again a vacuum of 50mbar was applied for 15min to evacuate the samples, afterwards a pressure of 5bar was applied for 30min. These cycles were also repeated once (Vac/Pres 1) or twice (Vac/Pres 2).

After the impregnation each specimen was thoroughly cleaned on the surface to remove excess impregnation solution and then dried either at ambient temperature (linseed oil) or at 90°C for two hours (tannin-based) followed by conditioning for 1 week in standardized climate. Weight was taken after conditioning.

### ***Assessment of magnetic behaviour***

To check the efficiency of the magnetite treatment, the samples were brought in contact with a permanent magnet and it was checked if they were able to bear their own weight at an angle of 80°, 90° and 180° (Fig. 5).

### ***Microscopic investigation***

Using a Nikon SMZ 1500 with 7.5 to 112.5 times magnification, micrographs of the samples cross section were taken. To give a visual clue for the presence of magnetite (this was important for the tannin-impregnated samples, as the colour differences were hard to spot) the samples were treated with 2M HCl

and 30% KSCN solution. The Fe<sup>3+</sup> ions in the magnetite form dark red Fe(SCN)<sub>3</sub> (iron thiocyanate) when in contact with the KSCN.

Zeiss Ultra Plus field emission scanning electron microscope (SEM) equipped with an annular backscatter electron and secondary electron detector (Carl Zeiss AG, Oberkochen, Germany) was used for further micrographs. Acceleration voltage was set to 5 kV and working distance was adjusted between 4 mm and 6 mm. Specimens were sputtered with gold prior to investigation.

## RESULTS AND DISCUSSION

### *Impregnation*

Using different concentration of magnetite in solution resulted in different shades of colour as expected. While for 2.5 and 5% magnetite content, the changes were almost not diminishable from impregnation without magnetite, the 10% solution resulted in a darker, almost black hue. It was much more noticeable for the oil-based system, as the tannin one is already brownish with a tendency to get darker with increasing tannin-concentration.

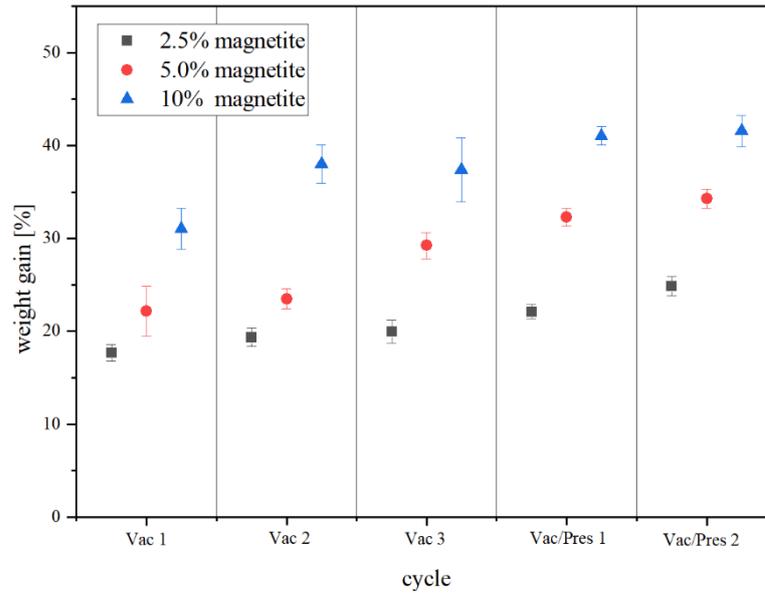
As expected, differences in weight gain and therefore impregnation efficiency can be observed for the two different wood species as well as the number and nature of impregnation cycles. Results for poplar samples are listed in Table 1 (standard deviation in brackets).

*Table 1: Weight gain of poplar wood samples in %*

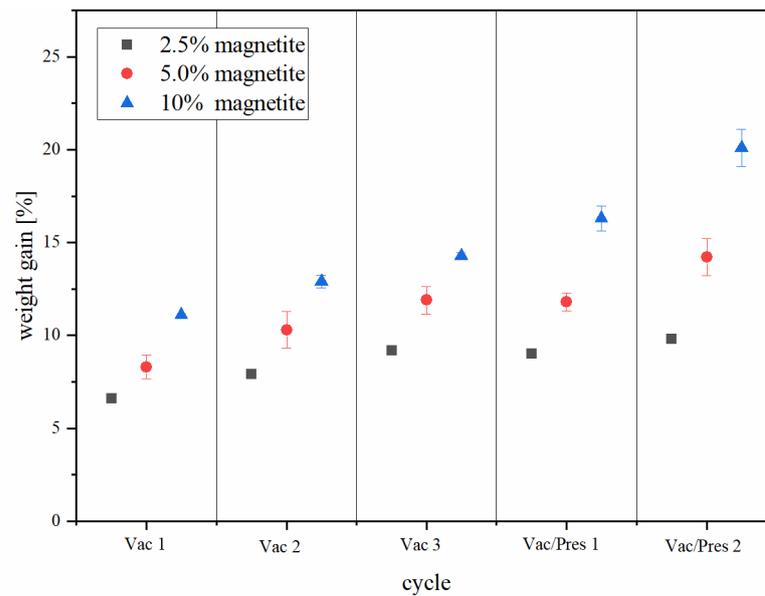
Specimen	Magnetite 2.5%		Magnetite 5%		Magnetite 10%	
	Linseed oil	Tannin	Linseed oil	Tannin	Linseed oil	Tannin
Vac 1	50.16 (2.038)	15.5 (0.097)	53.03 (1.106)	18.2 (0.523)	55.21 (1.664)	23.4 (0.826)
Vac 2	59.71 (1.679)	16.8 (0.138)	61.96 (2.304)	20.8 (0.394)	72.04 (0.964)	25.6 (0.336)
Vac 3	62.12 (2.225)	18.5 (0.113)	65.38 (1.963)	20.9 (0.117)	84.42 (1.123)	29.3 (0.168)
Vac/Pres 1	99.86 (1.993)	18.2 (0.173)	102.38 (2.997)	22.9 (0.956)	112.63 (3.368)	31.5 (0.934)
Vac/Pres 2	102.65 (3.687)	19.8 (0.113)	105.36 (3.024)	25.2 (0.353)	115.27 (2.049)	35.5 (0.147)

In general, the weight gain of poplar was much more noticeable as the one of beech as well as the differences between the different impregnation cycles when using hydrophobic magnetite/linseed oil. While with vacuum impregnation and 5% magnetite in solution a maximum weight gain of about 65% is possible for poplar, this value is almost doubled for vacuum/pressure impregnation (105%). The average weight gain for beech is shown in Fig. 1 for the linseed oil system. It's evident that the specimens are mostly saturated with impregnation solution after the first cycle (either vacuum or vacuum/pressure) as there is only a small of around 2% from one vacuum cycle to three vacuum cycles.

As soon as uncoated magnetite in a tannin solution is used to impregnate beechwood samples, a couple things need to be considered. (i) Tannin does form complex with different metals and metal oxides (ii) agglomeration of particle can occur (iii) stability of the suspension. The first concern can be dealt with, by preparing the impregnation solution fresh and use immediately after mixing. Agglomeration and dispersion of the magnetite nanoparticles within the solution can be assured by vigorous mixing/stirring of the solution. As shown in Fig. 2 an increase of around 5% in weight gain can be observed when repeating the vacuum phase multiple times. The particles or the particle/tannin agglomerations might block the vessels a few seconds after the vacuum is released and therefore hinder more particles from entering the samples. If the vacuum is applied a second or third time, these blockages will eventually be removed from the cross-section allowing more magnetite into the wood and therefore increase the loading. This effect can also be observed for the combined vacuum/pressure cycle where the second iteration allows a weight gain of up to 8% of the maximum magnetite concentration.

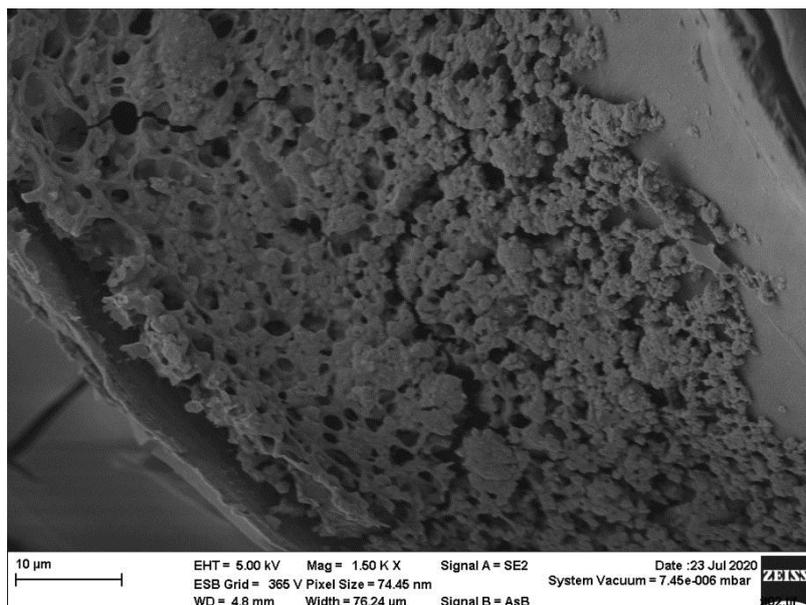


**Figure 1: Weight gain of beech samples after impregnation with linseed oil/magnetite solution**



**Figure 2: Weight gain of beech samples after impregnation with tannin/magnetite solution**

The SEM micrograph in Fig. 3 shows the equal distribution of magnetite within poplar wood sample using linseed oil as an impregnation medium. A homogeneous distribution of the magnetite even far away from the edges can be observed, which explains (i) the higher increase in weight when using linseed oil as carrier medium (at least to some extent) and (ii) the better results for magnetic behaviour compared to the tannin-based system (Table 2).



*Figure 3: Scanning electron micrograph of poplar and linseed/magnetite*

Fig. 4 shows the results of the KSCN stain for visual microscopy for beech with 2.5% magnetite in tannin. It is clearly visible that the majority of the iron oxide is stored near the edge of the specimen as the reddish colour tends to get lighter with increasing longitudinal distance from the edge. This supports once more the theory that bigger chunks of magnetite tend to clog the vessels near the edges. Staining for the linseed-oil impregnated samples took a little bit longer as the particles were all covered in oil and oleic acid. These samples did show a much more homogeneous distribution of magnetite within the specimens as they were able to infiltrate the wood easier due to not forming complexes and therefore bigger chunks with the impregnation medium.



*Figure 4: Comparison of KSCN stained (left) and unstained (right) beechwood with magnetite*

### ***Magnetic properties***

Goal of this procedure was to determine if the samples were able to bear their own weight when subjected to a paramagnet. Trials were done at three different angles and the following evaluations were possible (-) doesn't stick to the magnet, (o) moves in the beginning, but sticks, (+) good grip, doesn't move. Results for Vac/Pres 1 are shown in Table 2. The tannin-based system works only at a high concentration (10% for beech, and 5+% for poplar), this once more confirms that not as much magnetite is fixed in the tannin system compared to the oil-based one. Additionally, the amount of magnetite present in the wood is too little for the combination tannin-solution/beech wood, to give satisfying results.

Generally, poplar was much easier to impregnate as already shown in the weight gain. This, in combination with the lower density of poplar compared to beech wood, made it more likely to stick to the magnet. Additionally, it was possible to stack multiple pieces of impregnated poplar wood onto one another using a magnet at an angle of 80° (see Fig. 5).

**Table 2: Evaluation of the magnetic behaviour**

angle	Beech						Poplar					
	Linseed oil			Tannin			Linseed oil			Tannin		
	2.5	5	10	2.5	5	10	2.5	5	10	2.5	5	10
80°	+	+	+	-	-	o	+	+	+	-	o	+
90°	o	+	+	-	-	-	+	+	+	-	o	+
180°	o	o	+	-	o	+	+	+	+	+	+	+



**Figure 5: poplar and linseed oil-based magnetite system (left), tannin-based magnetite system (right)**

## CONCLUSION

In this work it was shown, that it was possible to impregnate beech and poplar with magnetite nanoparticles using either a water- or an oil-based system. Due to the tendency of tannin to complex with metal oxides, solutions have to be prepared fresh immediately before use. Although multiple vacuum cycles show a small change in the degree of impregnation, a combination of vacuum and pressure delivers much better results (up to twice as much weight gain). Preparing the magnetite nanoparticles ex-situ has shown the big advantage, that not the whole piece of wood has to be submerged in iron salt solution followed by ammonia, as shown in other studies, which makes this treatment also applicable for species that react with ammonia. The oil-impregnated samples did show the characteristic orangey taint that is associated with linseed oil. At magnetite concentrations of 2.5 and 5% almost no differences could be observed in the appearance, while with 10% magnetite the colour shifted to a darker range. The same is true for the tannin-based system. Although it was possible to render the samples magnetic with the tannin-based system, results for linseed oil are much more promising. Furthermore, this system is able to cure at ambient temperature while the tannin one has to be heated to 90°C for a certain time. Generally, the less dense poplar wood shows a better uptake of the magnetite and thus also works better with tannin/magnetite solution as beech does. Impregnation of the two wood species with 5% magnetite in linseed oil gives satisfying results for both, vacuum and vacuum/pressure procedure. If the samples are e.g. laser-engraved with your logo they can be used as giveaways at exhibitions or similar.

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## Green method of hardwood residue functionalization for obtaining biocomposites

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**Keywords:** Ball milling, hardwood sawdust, mechanochemical ammoxidation, wetting properties, wood-plastic composite

### ABSTRACT

The ammoxidation method of hardwood particles functionalization, despite its efficiency, proceeds for a long time and requires the enhanced consumption of the reagents. The study focused on a study of the effect of the mechanochemical ammoxidation of grey alder sawdust as a filler on its compatibility with recycled polypropylene (rPP) in wood-plastic composites (WPC). The mechanochemical treatment was carried out using a planetary ball mill with different rotational speed and a modification time of 0.5 h. With growing rotational speed from 0 to 600 rpm, the nitrogen content in sawdust was enhanced from 0.3% to 3.4% that was accompanied by both oxidative destruction and polymerization/condensation processes. The obtained WPC samples filled with the sawdust microparticles mechanochemically ammoxidized at the optimal conditions were characterized by better mechanical properties and hydrophobicity compared to the composites containing the microparticles functionalized by the ordinary ammoxidation method.

### INTRODUCTION

The mechanochemical treatment represents solid-phase chemical processes, initiated by the mechanical action in the presence of chemical reagents. This method is often used for modification of high molecular compounds, because it is more environmentally friendly and cost-effective compared to the existing ones. In contrast to chemical methods, in which heat energy is needed for initiating a reaction, the mechanochemical method allows an essential reduction in energy consumption and carrying out modification in a much shorter time while using compact reaction facilities. Mechanochemical reactions can be performed by milling, grinding or shearing in the absence of organic solvents and, they practically have no by-products or wastes. Taking the above mentioned into account, mechanochemical treatment complies with the requirements of “green” chemistry. Such a type of the treatment of wood and its products is often used in the pulp and paper industry ((Hon and Srinivasan 1983, Razumovskii et al. 2011). The reactions, proceeded in lignocelluloses during the treatment, have a radical nature. The initiation stage includes the cleavage of glycosidic bonds in hemicelluloses and phenol hydroxyl groups’ activation in lignin with phenolic hydrogen atom abstraction. The formed alkoxy and alkyl radicals are very unstable and react readily with oxygen to produce peroxy radicals ((Roffael et al. 2001, Karinkanta et al. 2013). Owing to the efficiency, the quantitative yield of a product and the simplicity of modification, the ammoxidation method for wood functionalization can be considered as one of the promising ones. Earlier, we have shown that the ammoxidized wood microparticles can be used as a compatible filler for obtaining WPC (Shulga et al. 2019). In spite of the functionalization efficiency, the ammoxidation treatment of the wood particles proceeded for more than 120 h and required the enhanced consumption of reagents. The aim of the research was to study the effect of the mechanochemical treatment of grey alder sawdust in the presence of ammonium hydroxide and persulphate ammonium on its usage efficiency in the wood-plastic composite (WPC) in comparison with the case of the sawdust functionalized by the ammoxidation in the absence of the mechanical treatment.

## EXPERIMENTAL METHODS

Grey alder sawdust was used for obtaining a filler. With this purpose, sawdust was treated by the following modes: 1) acid hydrolysis, milling followed by ammoxidation and 2) mechanochemical ammoxidation followed by milling. The hydrolysis was carried out with 0.1% HCl solution at 60°C for 5 h and a mass ratio of wood/water equal to 1/20. The ammoxidation of the hydrolysed sawdust microparticles (< 100 µm) was carried out during 120 h by their treatment with the NH<sub>4</sub>OH solution in the presence of (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (3.8 g per 1 g of air-dried wood) at a hydromodulus (wood/ NH<sub>4</sub>OH solution) of 1/50. The content of a fixed nitrogen (N) in the ammoxidized sawdust sample was 2.35%. The mechanochemical treatment was carried out in a planetary ball mill (Retsch, Germany) with zirconia balls. Technological parameters of the treatment were: rotational speed – 300-600 rpm, duration – 30 min. This ammoxidation was performed with the NH<sub>4</sub>OH/(NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> reaction mixture at an O/NH<sub>3</sub> mass ratio of 0.2 (1.5 g (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>/2.50 ml NH<sub>4</sub>OH per 1 g of the air-dried wood). After the treatment, the ammoxidized sawdust was washed to a neutral medium, dried and then milled. The functionalized sawdust particles were characterized by the elemental and wood polymers composition according to the classical chemical wood analysis. The fractional composition of the sawdust was determined by the sieve method. For the characterization of the morphology and shape of the treated sawdust particles, scanning electron microscopy (SEM) (Tescan 5136, Czech Republic) and transmission electron microscopy (TEM) (Leo-912 AB OMEGA) were used. Recycled polypropylene (rPP) (0.9 t m<sup>-3</sup>, 5.2/10 min at 230°C, 2.16 kg) was used as a WPC polymer matrix. The WPC samples were prepared from rPP and the sawdust microparticles, using a twin screw extruder and a moulding machine (HAAKE MiniLab II with MiniJet II, Thermo Scientific “HAKKE”) at a temperature of 175-180°C and a pressure of 600 bars. The microparticles content in the composites was 30%. Contact angle (CA) for the wood particles and the composite samples were measured in deionized water using a Kruss K100 tensiometer by the Washburn method and the Wilhelmy method, respectively. The total surface free energy and its dispersive (Lifshitz-van der Waals interactions) and polar (Lewis acid-base interactions) components were calculated using the Owens-Wendt-Rabel-Kaelble method. Adhesion energy relative to water was calculated according to the Young-Dupre equation. Mechanical tests for the composite samples were carried out according to ASTM D638 and EN ISO 178, using a universal machine “Zwick” (Germany).

## RESULTS AND DISCUSSION

The mechanochemically ammoxidation of the sawdust led to complex physicochemical conversions in the lignocellulosic matrix. As a result, at the activation speed of 300 rpm, the content of fixed nitrogen (N) in the sawdust microparticles dramatically grows from 0.3% for the initial sawdust to 2.0% for the ammoxidized one (Fig. 1). This was the most pronounced increase in the N fixation (almost 6.6 times) compared to the initial N content. The fixation of nitrogen is accompanied with lignocellulosic matrix oxidative destruction that is testified by the moderate yield (~ 87.5%) of the ammoxidized particles (Fig. 2) as well as by a decrease in the content of hemicelluloses. The mechanochemical ammoxidation was accompanied by increasing the relative content of cellulose and decreasing the content of hemicelluloses. The FT-IR spectrum of the sawdust, ammoxidized at a speed of 300 rpm, indicated the decrease in the content of hydroxyl groups, including phenolic hydroxyl ones, and methoxyl groups as a result of their participation in the radical reactions, compared to the initial sawdust spectrum. The oxidative destruction of the lignocellulosic matrix at the ammoxidation was also expressed by the decrease in the mechanical strength of the functionalized sawdust particles. According to Fig. 3, the content of the particles < 100 µm in the fractional composition of the sawdust, modified at a speed of 300 rpm, was enhanced 12.5 times compared to the case of the initial residue.

With increasing the rotational speed of the ball mill from 300 rpm to 600 rpm, the yield of the ammoxidized particles dropped moderately, herewith the content of the fixed nitrogen increased from 2.0% to 3.4% (only 1.7 times). The increase of the speed led to the pronounced oxidative destruction of the lignocellulosic matrix, the participation of the matrix's oxidized groups in the formation of secondary covalent bonds, including oxygen- and nitrogen-containing groups, as a result of the complex radical conversions.

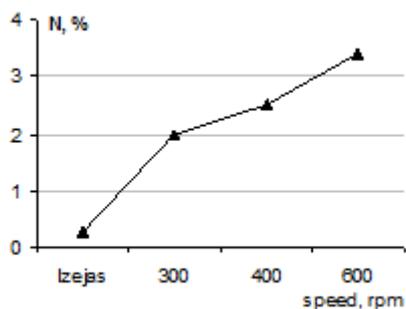


Figure 1: Content of fixed N in the modified sawdust

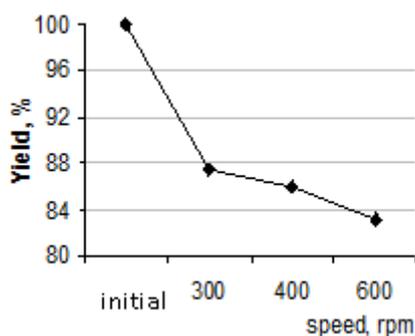


Figure 2: Yield of the modified sawdust

In a speed range of 400-600 rpm, simultaneously, with a dramatic decrease in the content of hemicelluloses and an increase in the relative content of cellulose, the polymerization/condensation processes in the ammoxidized lignocellulosic matrix manifest themselves most clearly. The released lignin radicals, located at the treated sawdust surface, actively participate in the radical processes, performing the function of a “glue”. This is expressed by a two-time reduction in the content of the microparticles < 100 μm in the sample, ammoxidized at 600 rpm, after its short-time milling, compared to the milled sample ammoxidized at 300 rpm (Fig. 3). The new covalent bonds formed during the radical transformations and the polymerisation/condensation processes positively effected on the sawdust hydrophobicity. The enhanced hydrophobicity was confirmed by the decrease of the particles’ zeta potential (in absolute values) from - 29.3 mv for the initial sawdust to - 26.8 mv for the ammoxidized sample and by the increase of a contact angle from 83.4° for the initial residue to 86.5° for the sawdust particles, modified at 400 rpm. The microparticles, mechanochemically modified at 400 rpm with a 2.5% N content, were used for obtaining rPP-based WPC. The TEM image of the used ammoxidized sawdust particle is given in Fig. 4.

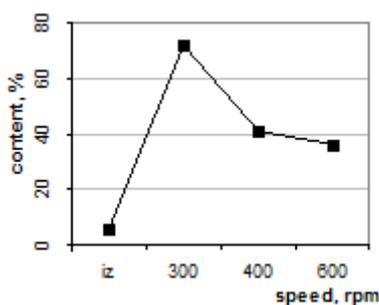


Figure 3: Content of the fine particles, depending on the speed

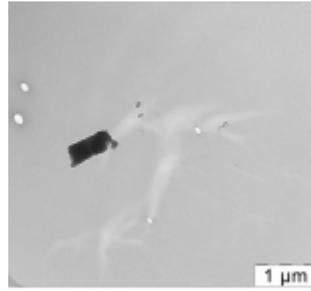


Figure 4: TEM image of the modified particle

The mechanical properties of the rPP-based composites filled with the initial, ammoxidized and mechanochemically ammoxidized hardwood particles with a filling of 30% are shown in Table 1. According to the data, the WPC samples filled with the functionalized sawdust, both chemically and mechanochemically, are characterized by an essential grow in the mechanical strength in comparison with the sample with the initial sawdust. The tensile strength at break and bending strength of the composites with the functionalized sawdust particles increase by 23-30% and 22-32%, but tensile and bending modulus of rupture is enhanced by 18-27% and 18-36%, respectively. Simultaneously, the deformability of the composites decreases, namely, elongation at break and deflection drop by 37-58% and 36-63%, respectively.

Table 1. Mechanical properties of WPC samples

Filler	Tensile strength, [MPa]	Tensile modulus [MPa]	$\epsilon_T$ ,* [%]	Bending strength, [MPa]	Bending modulus, [MPa]	$\epsilon_B$ ,** [mm]
Initial	21.1±0.4	615±14	14.4±0.5	24.8±0.5	1635±28	7.8±0.3
Ammoxidized	26.3±0.5	740±16	10.5±0.3	30.3±0.8	1935± 32	5.7±0.4
Mechanochem. ammoxidized	27.5±0.5	784±17	9.1±0.5	32.8±1.0	2230±34	4.8±0.4

\* $\epsilon_T$  - elongation at break, \*\* $\epsilon_B$  - deflection

At the same time, the WPC sample filled with the mechanochemically ammoxidized sawdust is characterized by better mechanical parameters than the composite with the initial sawdust. The characteristics of the wetting properties of the WPC samples are given in Table 2.

Table 2. Characteristics of the wetting properties of the WPC samples (30% filling)

Specimen	CA [degree]	Adhesion work [mN/m]	Dispersive part [mN/m]	Polar part [mN/m]	Free surface energy [mN/m]
Initial	96.5	64.1	25.8	1.3	27.1
Ammoxidized	99.9	60.3	27.5	0.6	28.5
Mechanochem.ammoxidized	101.4	58.4	28.1	0.4	29.1

It is seen that both the functionalization methods decrease the wetting properties of the WPC samples, accompanied by the increase in their contact angles, the decrease in their adhesion work and polar part of free surface energy in the comparison with the WPC sample filled with the initial sawdust. A higher contact angle of the WPC sample containing mechanochemically ammoxidized sawdust relatively to the composite with the chemically ammoxidized particles may be caused by the higher hydrophobicity of the mechanochemically functionalized sawdust particles.

## CONCLUSIONS

The mechanochemical ammoxidation of grey alder sawdust at a speed of 400 rpm leads to the increase of the mechanical properties and the decrease of the wetting properties of the WPC samples compared to the composite containing the sawdust ammoxidized in the absence of the mechanical action. Taking into

account that the ammoxidation in the ball mill takes an order of magnitude less time and lower consumption of ammonium persulfate than the ordinary ammoxidation for the introduction of the same nitrogen content in the lignocellulosic matrix, the mechanochemical ammoxidation can be considered as a hopeful way of a wood filler functionalization for obtaining WPCs.

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## Impact of modification by caffeine on some surface properties of beech wood

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**Keywords:** caffeine, wood modification, beech, colour, contact wetting angle, artificial aging

### ABSTRACT

Important aesthetic and functional surface characteristics of wood can be significantly influenced by modification with various impregnating solutions. Changes in colour and gloss are an aesthetic criterion, but the contact angle of wetting and the change in surface free energy can significantly affect the subsequent treatment of wood with a coating. Beech wood is typical with its low durability against fungi and insects. Resistance of beech wood can be increased using more environmentally friendly procedures. One of such ways is the utilisation of caffeine. In this work was investigated the effect of dipping of beech wood in caffeine solution and its effect on the change of gloss, colour, contact wetting angle and surface free energy. The sapwood zone of beech lumber was used to prepare the test specimens. Test specimens 40x40x10 mm were divided into 3 groups. The first group of test specimens was treated with 2% caffeine solution, the second group of samples was dipped in distilled water, the third group of test specimens was the reference. Furthermore, the impact of artificial accelerated weathering in Xenotest was assessed. Modification of wood with caffeine and wood dipping have only moderate impact on the surface free energy and water contact angle. No significant effect was confirmed for the remaining properties.

### INTRODUCTION

Beech wood (*Fagus sylvatica*, L.) has good mechanical properties (Požgaj et al. 1993), but a very low resistance against wood-destroying fungi damage (EN 350). Traditional synthetic biocides are mainly used to increase bio-resistance at present (Reinprecht 2016). However, several papers examine non-traditional methods using various substances of natural origin, which can be expected to have a reduced impact on the environment (Reinprecht 2016).

One of the promising substances is also caffeine which biocidal effect against wood-destroying fungi and moulds has been confirmed in older and new works (Arora and Ohlan 1997, Ratajczak et al. 2018).

The appearance of wood, especially its colour, can be significantly changed by modification with a biocide. At the same time, wood in its original native state has a demonstrably positive effect on the human psyche (Ikei et al. 2017) and therefore it is desirable to keep it unchanged.

Treatment of wood by impregnation or modification can significantly change other surface characteristics, especially the contact angle of wetting with water and surface free energy (Pánek et al. 2019). This effect can affect the quality of the subsequent surface treatment with coating systems or gluing (De Meier 2005, Pizzi and Mittal 2011).

The aim of this experimental work is to determine the changes in selected surface characteristics of beech wood caused by treatment using dipping in caffeine solution. Furthermore, the influence of artificial accelerated weathering in the Xenotest on changes of colour, gloss and surface wetting of treated and untreated wood is evaluated.

#### **Material and methods**

Beech wood (*Fagus sylvatica*, L.) with a mean oven-dry density  $\rho_0 = 678 \text{ kg.m}^{-3}$  (SD = 11,22  $\text{kg.m}^{-3}$ ) was used to prepare 24 test specimens measuring 40x40x10 mm (RxLxT). The surfaces were sanded with 120 grit sandpaper. The samples were divided into 3 groups: 1) Reference without treatment (B-Ref), 2) dipped in 2% caffeine (supplier - Sigma Aldrich) solution (B-Caf), 3) dipped in distilled water (B-H<sub>2</sub>O). The uptake of caffeine solution and distilled water during dipping was 120  $\text{kg.m}^{-3}$  (SD = 15.1  $\text{kg.m}^{-3}$ ). Before the further

experiments, the samples were conditioned at a temperature of  $T = 20\text{ }^{\circ}\text{C}$  and an air humidity of  $\phi = 65\%$  until an equilibrium moisture content was reached.

The colour of the samples was determined using the Spectrophotometer CM-600d (Konica Minolta, Osaka, Japan) and the parameters  $L^*$ ,  $a^*$ ,  $b^*$  were measured, from which the total colour change of the treated and the weathered wood was subsequently expressed during the experiment. according to the relationship Eq. 1.

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

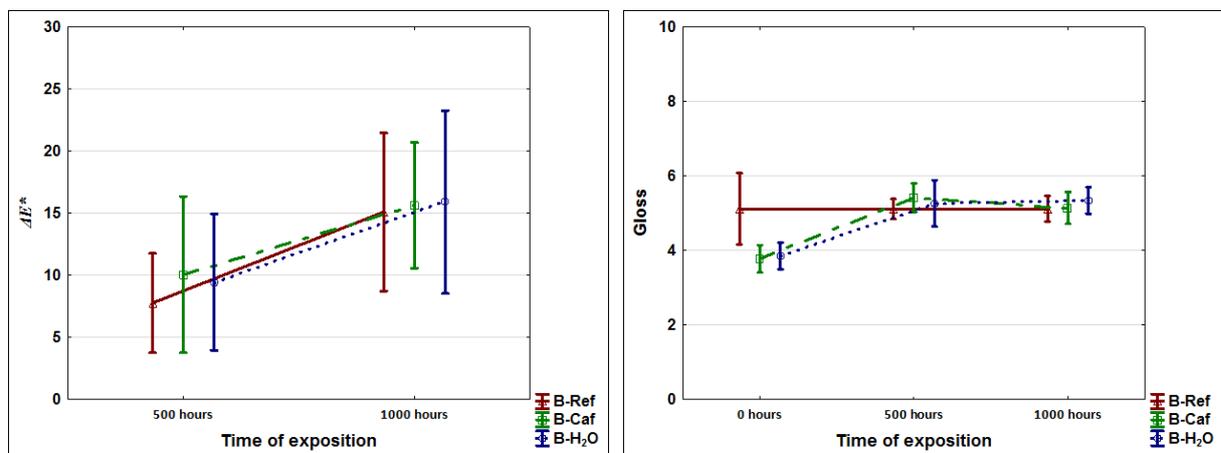
Gloss was evaluated using glossmeter MG268-F2 (KSJ, Quanzhou, China) under  $60^{\circ}$  in longitudinal direction. The contact angle of wetting with water ( $CA^{\circ}$ ) and surface free energy (SFE) were determined using a Krüss DSA 30E goniometer (Krüss, Hamburg, Germany).  $5\text{ }\mu\text{l}$  drops of distilled water were used for the determination of surface wetting and subsequently also diodomethane for the determination of SFE with software Krüss (Krüss, Hamburg, Germany) and ORWK model. Colour, gloss,  $CA^{\circ}$  and SFE were evaluated according to the methodology deeper described in Pánek et al. (2019)

All sets of the tested samples were subjected to artificial accelerated weathering in Xenotest Q-Sun Xe-3 (Q-Lab, Cleveland, OH, USA) with repeating steps 2.5 hour of irradiation exposure and 0.5 h of spray with distilled water. Temperature of air was  $45\text{ }^{\circ}\text{C}$ , temperature at black panel  $80\text{ }^{\circ}\text{C}$ , Air humidity  $\phi = 30\%$ , TUV =  $55\text{ W}\cdot\text{m}^{-2}$ , Total energy during 1000 hours of test  $167\,530\text{ kJ}\cdot\text{m}^{-2}$ . Weathering in the Xenotest better imitate the exterior conditions compared to a UV chamber with fluorescent lamps, as confirmed also by the manufacturer of Xenotest f. Q-Lab (U.S.).

Changes of colour, gloss, water contact angle and SFE were evaluated after dipping in caffeine solution and distilled water, as well as after 500 and 1000 hours of artificial accelerated weathering in the Xenotest. Sample surfaces were evaluated after weathering using a Lext Ols 4100 confocal laser scanning microscope (Olympus, Tokyo, Japan) at 108x magnification. Statistical analyses of the measured values were performed using software STATISTICA 13.

## RESULTS AND DISCUSSION

The colour changes of beech wood after caffeine treatment, soaked in distilled water and also during weathering in the Xenotest are shown in Fig. 1. According to the parameters  $L^*$ ,  $a^*$ ,  $b^*$ , beech wood is one of the lighter species occurring in Central Europe (Oltean et al. 2008). It is clear (Fig. 1) that neither caffeine treatment nor soaking in water had a significant effect on total colour changes. The total colour difference after soaking in caffeine was  $\Delta E^* = 0.46$  and after soaking in distilled water  $\Delta E^* = 0.42$ , which are unobservable changes with the naked eye.



**Figure 1: Colour (left) and Gloss (right) changes of beech wood after treatments and artificial accelerated weathering in Xenotest. (Mean; Whisker: Mean $\pm$ 2\*SD; n=16).**

More significant colour changes occurred only due to weathering in the Xenotest, where the irradiation causes photodegradation of lignin and water spraying its leaching (Volkmer et al. 2013). However, the observed differences between all types of samples tested were negligible. From the results shown in Fig. 1, it is clear that the treatment of beech wood with caffeine has no significant effect on changes in its colour. The gloss changes are also documented in Fig. 1. After treatments using dipping in caffeine and water, there was a slight decrease in gloss observed, but during weathering in the Xenotest these differences decreased. Due to the fact that the gloss of the wood was small and the surface can be characterized as matt, the differences recorded are negligible from the point of view of an external observer.

SFE changes are listed in Tab. 1. It can be seen that the total SFE has not changed significantly, only small increase was observed after dipping, and is in the range of works cited results for beech wood (De Meier 2005). However, the Polar and Disperse ratios components of SFE have changed. Probably by simultaneous leaching of water-soluble beech wood extractives and also by chemical change of surfaces due to caffeine treatment. This predicts that there could be a change in wetting as well as adhesion of coatings to the caffeine-treated wood. As the contact angle of wetting with water was reduced, especially water-borne paints could be positively affected (Tab 1).

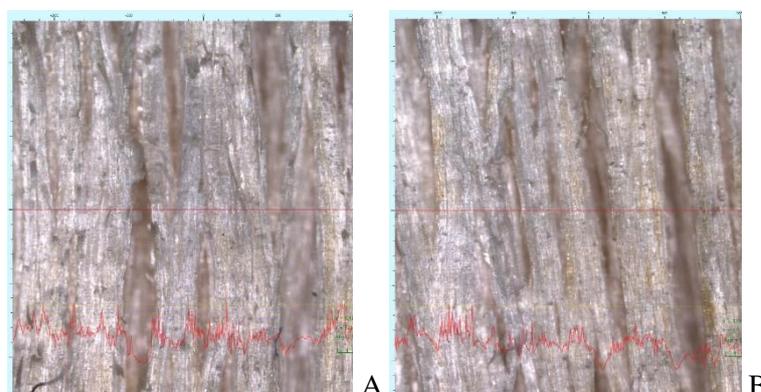
**Table 1: Surface free energy of beech wood after treatments and water contact angle during artificial accelerated weathering in Xenotest**

Type of samples	Surface free energy [Mean ± SD in mN.m <sup>-2</sup> ]			Contact angle of water [°]		
	Total	Disperse	Polar	0 hours	500 hours	1000 hours
B-Ref	49,15±4,67	41,21±2,04	7,94±2,63	63.7 (8.3)	0.0 (0.0)	0.0 (0.0)
B-H <sub>2</sub> O	53,84±6,91	37,7±2,82	16,14±4,08	57.8 (5.0)	0.0 (0.0)	0.0 (0.0)
B-Caf	53,7±3,99	42,46±2,08	11,24±1,91	52.6 (8.7)	0.0 (0.0)	0.0 (0.0)

Mean (SD); Number of measurements n = 20

During weathering in the Xenotest, the wood surfaces degraded very fast, which is documented in Fig. 2 using confocal laser scanning microscope. By leaching of the photodegraded lignins, the individual wood fibres separated and microcracks were formed. The surface of the photodegraded wood consists mainly of cellulose (Volkmer et al. 2013) and for this reason CA ° water was already 0 ° after 500 hours of weathering, because there was an immediate infiltration of the deposited drop. The result points to the low resistance of beech wood to weathering, which, in addition to its low bio-resistance, makes it difficult to use it in extreme outdoor applications.

The caffeine treatment itself did not have a positive impact from this point of view, caffeine is also leachable from wood by water according to the work of Ratajczak et al. (2018). It will therefore be necessary to use additional barrier protection with coating systems protecting the surfaces of beech wood against the weathering (Reinprecht 2016).



**Figure 2 : B-Caf (A) and B-Ref (B) - after 1000 hours of weathering in Xenotest – possible to see microcracks creation and fibrils respectively cells release associated with the increase in roughness.**

## CONCLUSIONS

The effect of caffeine treatment on changes in selected surface characteristics of beech wood was investigated in this experiment. Dipping in caffeine solution did not have a significant negative effect on the change in gloss and colour of the wood, so the overall appearance for the outside observer will remain unchanged after treatment. Some effect on changes in SFE and water wetting contact angle was observed. As the caffeine treatment itself did not have a positive effect on wood surfaces during artificial accelerated weathering in the Xenotest, additional protection by coating systems will be necessary. Reducing the contact angle of wetting with water suggests that the treatment of beech wood with this biocide of natural origin can positively affect the adhesion and overall durability of exterior waterborne coating systems.

## ACKNOWLEDGMENTS

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## Drying schedules for thin oak boards and blanks

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**Keywords:** wood drying, oak wood, drying schedule, mass transfer coefficient, drying stresses, mathematical simulating

### ABSTRACT

In recent decades, a constant increase in the price of valuable wood species has taken place. Largely due to this, multilayer wood products are becoming increasingly popular. In them, the outer layers are made of valuable hardwood of not large thickness, and the inner layers are made of cheaper coniferous wood. Having high consumer properties, such wood products are much cheaper. At the same time, they are usually more stable in operation than products made from expensive solid wood.

Thin blanks are produced by longitudinal cutting of dry wood (so-called sawn veneer) or wet wood, followed by drying. In addition, wet blanks up to 8-10 mm thick and even thicker (thick veneer) can be produced using modern longitudinal slicing machines.

Until recently, there was no description of the schedules specially designed for drying boards and blanks thinner than 19 - 22 mm in the technical literature. There are only a few recommendations to try using well-known schedules for drying very thin hardwood lumber.

Six drying schedules of thin oak boards and blanks, as well as thick sliced veneer with a thickness of 16 mm to 19 mm, 13 mm to 16 mm, 10 mm to 13 mm, 8 mm to 10 mm, 6 mm to 8 mm and less than 6 mm are described in this paper. The schedules were developed on the basis of mathematical simulating of the stress-strain state of wood during multistage convective drying and the results of laboratory testing experiments. Recently obtained new data on mass transfer coefficients made it possible to more accurately calculate the drying stresses in wood primarily at the initial stage of the drying process.

An experimental validation of the developed schedules showed a reduction in drying time depending on the thickness from 5 to 18% compared with the known ones for sawn timber 19 mm thick. Schedules provide good drying quality and complete preservation of the natural properties of oak wood.

### INTRODUCTION

Oak is one of the most popular hardwoods in Europe. Its wood is highly valued for its excellent consumer properties: beautiful appearance, durability, high strength and hardness, good workability, resistance to decay. Oak wood is widely used for the manufacture of furniture, doors, windows, stairs, decorative items, etc. as well as block parquet and parquet boards.

Several varieties of oak grow in Europe. In Eastern Europe, the pedunculate oak (*Quercus robur* L.) is the most widespread. However, the reserves of this valuable wood are limited. Thanks to the high and stable demand for oak wood, its prices are permanently growing. For this reason, multi-layer products are becoming more and more popular. The outer layers in them are made of valuable hardwood of a small thickness, and the inner layers are made of cheaper coniferous wood. Having high consumer properties, such products are much cheaper. In addition, they are usually more stable in operation than products made of expensive solid hardwood.

Today thin blanks are produced by longitudinal cutting of dry wood (so-called sawn veneer) or wet wood, followed by drying. In addition, wet blanks up to 8-10 mm thick and even thicker (thick veneer) can be produced using modern longitudinal slicing machines.

Before latest, there were practically no schedules in the technical literature developed specially for drying wet hardwood blanks with a thickness of less than 19-22 mm. There are only a few recommendations to try to use known schedules for drying the thinnest hardwood lumber with a thickness of 22-25 mm (Boone et al. 1988, Pratt et al. 1997, Keey et al. 2000, Cividini 2000). If necessary, thin hardwood blanks are dried according to the schedules intended for thicker material, which unreasonably increases the duration of the process and negatively affects the cost of finished products.

Manufacturers of longitudinal veneer planers and dryers recommend to use conventional roller dryers for drying veneer up to 3 mm thick. If the veneer does not have time to dry in one pass, it is recommended to pass it through the dryer again. At the same time, modern machines make it possible to obtain veneer with a thickness of up to 10-13 mm. But in the technical instructions supplied with the dryers there is no information on how to dry such veneer. In practice, woodworkers have to repeatedly pass it through a roller dryer to dry thicker veneers. However, due to the lack of regulation, the relative humidity of the air is very low in such dryers. On the one hand, this speeds up the drying process, and on the other hand, it often leads to the formation of cracks. As a result, drying costs are high due to significant energy and labor costs. In addition, with this drying technology, wood often dries unevenly.

Drying schedules for thin oak blanks that are developed on the basis of laboratory experiments and mathematical simulating are described in this paper.

## MATERIALS AND METHODS

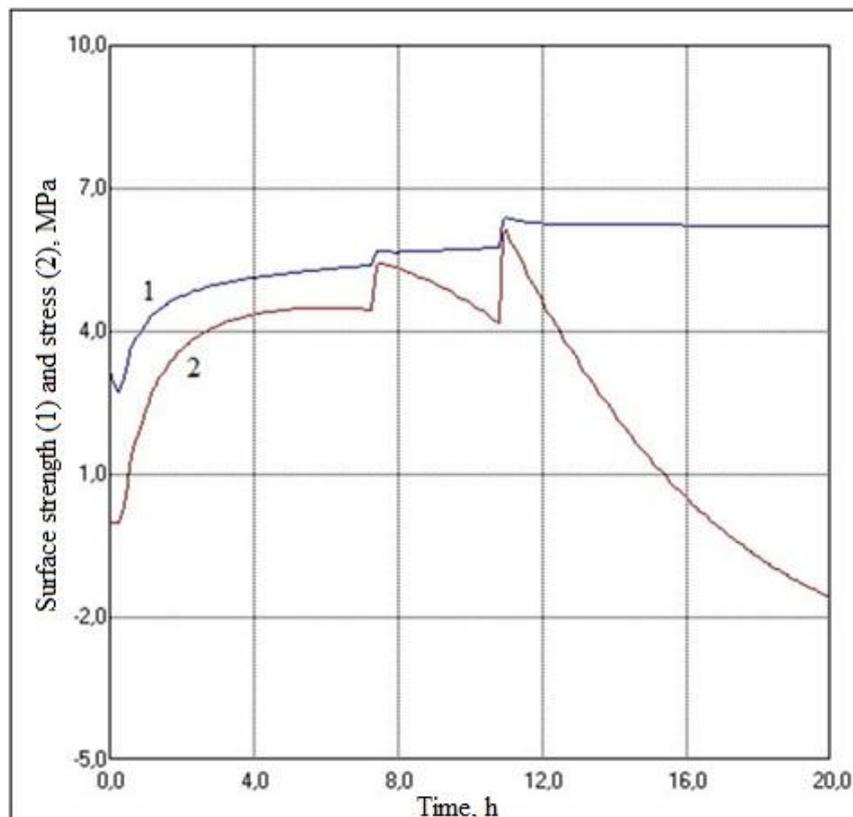
On the thickness as a determining dimension thin blanks and thick veneers occupy an intermediate position between ordinary veneers and sawn timber. Experimental studies of the dynamics and kinetics of drying of oak blanks with a thickness of 8, 10, 12 and 14 mm showed that, despite the small thickness, the Biot number for them is approximately the same as for coniferous sawn timber of normal thickness. The Biot number is the ratio of the moisture exchange coefficient to the moisture diffusion coefficient multiplied by half the thickness of the plate. The higher its value, the higher the moisture gradient in the surface zones of the material and consequently the level of stresses at drying. Analysis of numerous drying curves showed that the nature of the drying processes of thin oak blanks is similar to the drying of conventional sawn timber, but differs in the scale of time. The formation of surface cracks on several experimental samples indicates the need to regulate the air relative humidity when drying such material.

When determining the rational number of stages of drying schedules for thin oak blanks, it was decided to use the three-stage structure of drying schedules for sawn timber of conventional sizes like in Russian standard - GOST 19773-84. Also, the values of the first and second transitional wood moisture content were preliminary determined at the level of 35% and 25%, respectively. The temperature levels of the regimes were chosen taking into account the possible darkening of oak wood due to prolonged temperature exposure (Skuratov 2010).

To determine the optimal parameters of the drying schedules, a mathematical model simulating the stress-strain state of wood during the drying process was used (Ugolev and Skuratov 1992). Some changes have been made in the mathematical model. A nonlinear dependence of the shrinkage coefficient on wood moisture content (Ugolev et al. 1998). and refined dependences of the modulus of elasticity and ultimate strength of oak wood in tension across the grain on its moisture content and temperature were introduced (Skuratov 2008). On the basis of the results of recent studies (Skuratov et al. 2016, Skuratov 2018), the conditions of moisture exchange between wood and air at drying were precised. These changes made it possible to more accurately calculate the drying stresses in wood, especially at the initial stage of the process.

## RESULTS AND DISCUSSION

At the initial stage of simulating, the safe relative humidity of the air at which the drying stresses in the surface zone of blank of the given thickness, do not reach the tensile strength of oak wood was selected. When computing, oak wood was considered freshly cut with an initial moisture content of 80%. The parameters of the first stages of drying schedules were determined especially carefully, since the formation of surface cracks is most likely at the beginning of the process. Safe values of the relative air humidity for the second and third stages of the schedules were selected in the same way. Figure 1, as an example, illustrates the changes in the wood tensile strength and surface stresses during a three-stage drying of 10 mm oak blank with an initial moisture content of 80% at the following values of temperature (t) and relative humidity ( $\phi$ ): t<sub>1</sub> = 64 °C and  $\phi$ <sub>1</sub> = 0.71; t<sub>2</sub> = 71 °C and  $\phi$ <sub>2</sub> = 0.63; t<sub>3</sub> = 80 °C and  $\phi$ <sub>3</sub> = 0.34. As graph 1 shows, surface stresses increase rapidly at the start of drying. Then, during the transitions from step to step, they also increase abruptly, approaching the ultimate tensile strength, but do not reach it.



*Figure 1. Changes in the calculated ultimate strength of wood (1) and drying stresses (2) in the process of three-stage drying in the surface layers of 10 mm oak blank. Positive - tension stress, negative - compression stress*

The developed drying schedules for thin oak blanks and boards are presented below (Table 1). Schedules for 6 groups of oak wood thicknesses are coordinated by the average wood moisture content. Temperature (t), relative humidity ( $\phi$ ) and psychrometric difference ( $\Delta t$ ) are used as air parameters in the table. During of the experimental verification of the developed schedules 18 drying tests in which raw specimens with thickness of 8, 12 and 16 mm were dried to a moisture content of  $8 \pm 1\%$  were carried out. All drying runs were successful. None of them showed cracking of the samples and excessive unevenness in moisture content in thickness at the end of the drying process exceeding 1-1.3%. The minimum reduction in drying time in comparison with drying according to the schedule for blank with a thickness of 19 mm, depending on the thickness of the blanks, ranged from 5% to 18%.

**Table 1: Drying schedules for thin oak blanks and boards**

Average moisture content, %	Air parameters	Thickness of blank or board, mm					
		< 6	6 - 8	8 - 10	10 - 13	13 - 16	16 - 19
>35	t, °C	66	65	64	62	60	58
	Δt, °C	12	9	7	5	4.5	4.5
	φ	0.52	0.66	0.71	0.75	0.78	0.80
35-25	t, °C	77	74	71	68	65	63
	Δt, °C	16	12.5	12.5	8	7.5	6.5
	φ	0.46	0.55	0.63	0.67	0.69	0.71
<25	t, °C	80	80	80	80	80	80
	Δt, °C	22	22	22	22	22	22
	φ	0.34	0.34	0.34	0.34	0.34	0.34

## CONCLUSIONS

Recently obtained new data on moisture transfer coefficients have made it possible to improve the mathematical model and more accurately calculate the drying stresses in wood, primarily at the initial stage of the drying process.

Experimental verification of the developed modes has shown that they visibly reduce the drying time and provide good drying quality for thin oak blanks less than 19 mm thick while fully preserving the natural wood properties.

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## Bending moments study of upholstered furniture frames under light-service load by FEM

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**Keywords:** upholstered furniture frame, bending moment, Scots pine, PB/OSB/PLY, CAE/FEM

### ABSTRACT

A study has been made of the distribution of bending moments for light-service load of upholstered furniture frames by finite elements method (FEM). Two constructions of one-seat upholstered furniture frame with side plates and staple corner joints between rails and side plates were 3D modelled using CAD system - without (construction A) and with reinforcing details under the rails of the seat (construction B). Linear static finite elements analyses (FEA) were carried out with CAE system simulating light-service loading of the frames. The orthotropic material characteristics of Scots pine (*Pinus sylvestris L.*) for the rails and PB, OSB and PLY panels for the side plates are considered in the finite elements analyses. The laboratory derived coefficients of rotational stiffness of used staple corner joints were taken into account in the analyses. The distribution of bending moments in upholstered furniture frames with staple corner joints is presented and analyzed. It has been established that the maximum values of acting bending moments have been localized in the middle of the rear rail of the seat and in the joints of rear rail of the seat with side plates for both constructions A and B of the frame and for all used side plate materials. The results of this study can be helpful in strength design of upholstered furniture frames.

### INTRODUCTION

The size and distribution of bending moments in the skeletons of upholstered furniture are of particular importance for their strength. Strength design is an integral part of the process of upholstered furniture design. The strength study of the skeleton of upholstered furniture as a spatial structure with the help of modern computer programs on FEM gives the opportunity to get an idea of the actual complex load of structural elements and joints between them, as well as the whole structure.

Strength studies by FEM have been conducted by Smardzewski 1998 of chair side frames from pine wood and of a single-seat armchair made of wood (birch) and chipboard with staple joints (Smardzewski 2001) in order to optimize the materials used and the joints between component elements.

In Bulgaria, Marinova 1999, Kyuchukov and Marinova 1999, Marinova et al. 1999, Marinova and Genchev 2000 have been carried out study of internal forces distribution in solid wood chairs with different construction by FEM. They have been established the most loaded joint – the one between the rails and the back leg and the internal forces in this joint have been accepted as a strength criterion.

Kasal 2006 and Kasal et al. 2006 have been carried out strength investigations of three types of sofa frames constructed of solid wood (pine and beech) and wood based materials (OSB, PLY and MDF) with dowels and screw joints under seat and backrest loads by FEM. They have been established the moments and bending stresses distribution. In the result the authors have recommended to use wood composites instead solid wood in the production of upholstered seating furniture frames.

The purpose of this study was to establish the distribution of bending moments in upholstered furniture frames with side plates from PB, OSB and PLY and rails from Scots pine with a FEM based computer program simulating light-service loading of the frames.

## METHODS

Two 3D FEM models (construction *A* without and construction *B* with reinforcing details under the rails of the seat) of one-seat upholstered furniture frame with length 600 mm, width 725 mm and height 650 mm were created from pine solid wood (*Pinus Sylvestris* L.) rails 25×50 and side plates from PB, OSB and PLY – Fig.1. Mechanical characteristics of used materials are pointed in our previous publication (Staneva et al. 2018).

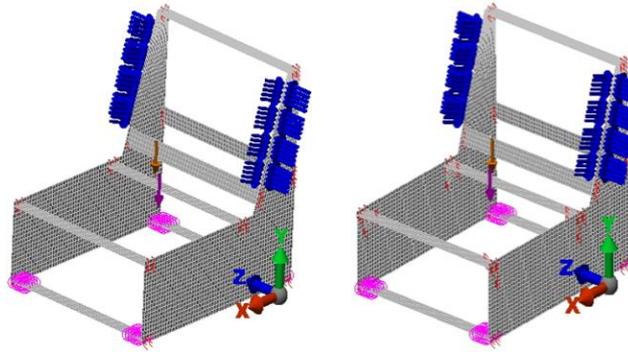


Figure: Upholstered frame constructions *A* and *B* - discrete models.

The generated mid-plane mesh has 5130 plate finite elements and 33616 DOF's for construction *A* and 5230 plate finite elements and 34096 DOF's for *B*. A linear static analysis of each 3D discrete model (*A* and *B*) was carried out with Autodesk Simulation Mechanical<sup>®</sup> by FEM. Orthotropic material type was introduced in the program. Semi-rigid connections between rails and side plates of the frame were simulated. The validity and more details of this approach are given in Staneva et al. 2018.

Each model was loaded with a total load of 800 N, 80% were set as a remote force with application point of 100 mm in front of the rear rail of the seat and 16% were distributed on the edges of the two sides of the backrest.

## RESULTS AND DISCUSSION

The results for bending moments are shown in Table 1, Table 2, Table 3 and in Fig. 2, Fig. 3 and Fig 4. Maximal values of acting bending moments  $M_{zz}$  are receiving in the middle of the rear rail of the seat (Table 1 and Fig. 2) for both constructions *A* and *B* and with slight differences for PB and OSB side plates and 1.12 times smaller for PLY side plates. A more even distribution of bending moments  $M_{zz}$  was established in construction *B*, relatively reducing its size in the middle of the seat rails compared to construction *A*. Maximal values of acting bending moments  $M_{xx}$  are received in the side plates – in the field of joining of rear rail of the seat and they are greatest for side plate from PLY, 2 times smaller for OSB and 2.3 times smaller for PB side plate – Table 2. The bending moments  $M_{xx}$  in the joints between the rear rail of the seat and the side plate in construction *A* are greater than those in construction *B*: 3.3 times for PB, 2.5 times for OSB and 2.6 times for PLY side plates (Table 1 and Fig. 4).

Table 1: Bending and twisting moments in the frame model for PB, OSB and PLY side plates.

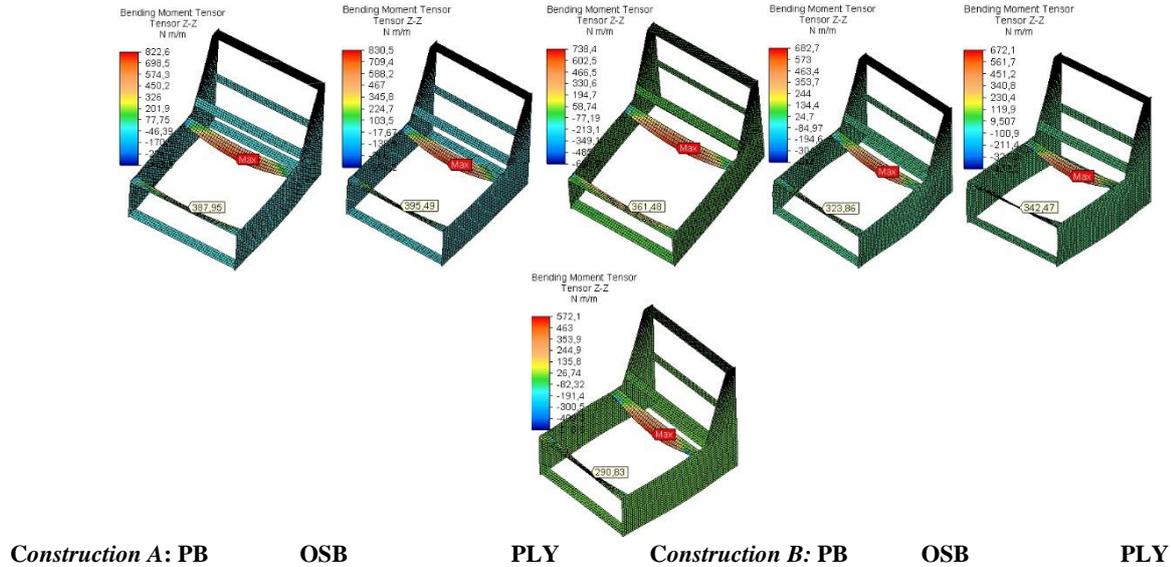
Parameters	Location	PB		OSB		PLY	
		A	B	A	B	A	B
$M_{xx}$ , [Nm/m]	side plate joint with rear rail	189.96	57.52	217.57	87.16	435.85	165.08
$M_{yy}$ , [Nm/m]	side plate joint with rear rail	95.35	-	103.68	-	137.7	-
$M_{zz}$ , [Nm/m]	rear rail – in the middle	822.62	682.71	830.54	672.11	738.0	572.08
$M_{xy}$ , [Nm/m]	side plate joint with rear rail	36.89	46.29	47.04	55.77	62.97	67.92
$M_{yz}$ , [Nm/m]	backrest joint with front rail	7.67	-	7.09	-	4.90	-
$M_{zx}$ , [Nm/m]	rear rail - joint with side plate	261.19	-	268.8	-	253.49	-

**Table 2: Bending and twisting moments in the side plates from PB, OSB and PLY.**

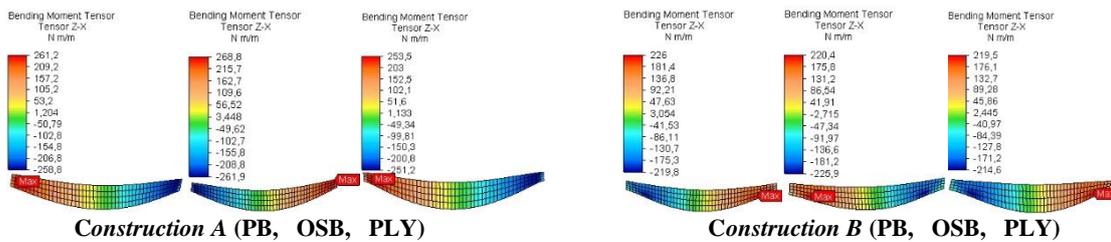
Parameters	Location	PB		OSB		PLY	
		A	B	A	B	A	B
$M_{xx}$ , [Nm/m]	rear rail joint	189.96	-	217.57	87.16	435.5	165.08
	rear rail – reinforcing elem.	-	57.45	-	-	-	-
$M_{yy}$ , [Nm/m]	rear rail joint	95.35	-	103.68	-	137.7	-
	rear rail – reinforcing elem.	-	87.48	-	58.74	-	100.07
$M_{zz} \cdot 10^{-9}$ , [Nm/m]	rear rail joint	4.87	1.05	3.73	9.54	8.89	2.51
$M_{xy}$ , [Nm/m]	rear rail joint	36.89	38.95	47.04	40.39	62.97	50.53
$M_{yz} \cdot 10^{-4}$ , [Nm/m]	rear rail joint	4.9	1.1	3.4	5.3	8.5	2.3
$M_{zx} \cdot 10^{-4}$ , [Nm/m]	rear rail joint	3.5	1.0	3.4	1.5	8.5	2.7

**Table 3: Bending and twisting moments in the rear rail of the seat for PB, OSB and PLY side plates.**

Parameters	Location	PB		OSB		PLY	
		A	B	A	B	A	B
$M_{xx}$ , [Nm/m]	joint with side plate	30.20	-	29.61	-	20.14	-
$M_{yy} \cdot 10^{-9}$ , [Nm/m]	joint with side plate	20.2	6.24	18.4	6.58	35.8	7.28
$M_{zz}$ , [Nm/m]	rear rail middle	822.62	682.71	830.54	672.11	738.40	572.08
$M_{xy} \cdot 10^{-4}$ , [Nm/m]	joint with side plate	20.6	13.7	20.5	13.3	20.6	14.0
$M_{yz} \cdot 10^{-4}$ , [Nm/m]	joint with side plate	29.2	6.1	15.9	6.9	46.8	10.3
$M_{zx}$ , [Nm/m]	joint with side plate	261.19	-	268.80	-	253.49	-



**Figure 2: Distribution of bending moments Z-Z in the fem model**



**Figure 3: Distribution of bending moments Z-X in the rear rail of the seat**

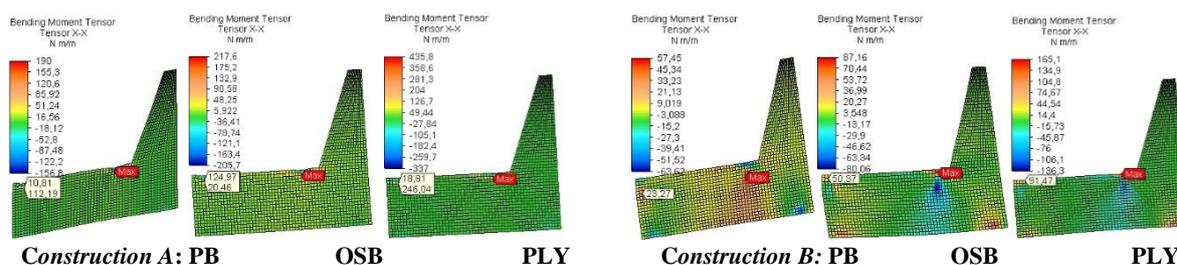


Figure 4: Distribution of bending moments X-X in the side plates for construction A and B

Maximal values of twisting moments  $M_{zx}$  are received in the rear rail of the seat in the fields of joining with the side plates for construction A and in the area above the reinforcing elements for construction B with a slight difference for the different materials of the side plates for both constructions – Fig. 3.

## CONCLUSIONS

Maximal values of the acting bending moments are received in the middle of the rear rail of the seat for both constructions A and B of the studied one-seat upholstered frames and for all materials of the side plates. These bending moments are almost equal for frames with PB and OSB side plates and about 2 % less for PLY. Maximal values of the acting twisting moments are received in the rear rail of the seat, in the fields of joining with side plates for both constructions A and B and are almost equal for the different materials of the side plates. This determines the joints between the rear rail of the seat and the side plates as the most loaded in the upholstered frame for both constructions and for all materials of the side plates.

From the studied two constructions - without (A) and with reinforcing details under the rails of the seat (B) of one-seat upholstered frames, the distribution of the bending and twisting moments in construction B is more favourable for its strength.

The bending and twisting moments calculated by FEM can also be used for strength design of elements, joints, as well as entire upholstered frames.

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## Selected structural, physical and acoustic properties of sycamore maple wood with fiddleback figure

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**Keywords:** wood, maple, fiddleback figure, anatomy, physical properties, acoustics

### ABSTRACT

The sycamore maple with a fiddleback figure (FB) and straight grained (C) was sampled to study structural properties, wood density and dimensional stability, in relation to elastomechanical and acoustic properties of wood. Test specimens with cross section of 30 by 30 mm were conditioned in a normal-, dry- (20 °C / 35 RH) and humid- climate (20 °C / 85 % RH). The study did not confirm structural differences between two categories of maple at the microscopic level. Wood density at similar growth ring width of both wood categories was comparable ( $\rho_C = 625 \text{ kg/m}^3$ ,  $\rho_{FB} = 627 \text{ kg/m}^3$ ), as well as their transverse dimensional stability. The mean wavelength in wavy maple has reached 9.0 mm (CV = 13.7%), however not detected at any of microscopic magnification. Ultrasound velocity in longitudinal and radial direction of two maple categories was similar. The lower sound velocity and therefore reduced mechanical stiffness of the maple with fiddleback figure was confirmed only in tangential direction ( $v_C = 1307 \text{ m/s}$ ,  $v_{FB} = 1165 \text{ m/s}$ ). The damping of sound along the grain was low at both, straight grained and wavy maple, and increased at reduced wood stiffness. No significant difference was found also in the bending strength of both types of wood.

### INTRODUCTION

The fiddleback figure in sycamore maple wood (*Acer pseudoplatanus* L.) is a known phenomenon, with studies on its biological origin, genetics, the formation as well as its anatomical, physical, mechanical and decorative characteristics (Alkadri et al., 2018; Bucur, 2006; Ewald & Naujoks, 2015; Harris, 1989; Spycher et al., 2008). This decorative specialty is highly valued in the manufacture of wooden musical instruments, unique furniture, and in the production of sliced veneer (Krajnc et al., 2015). In the making of violins, back plates, ribs and necks are generally constructed of maple wood – meeting parameters just opposite to spruce wood (Kudela & Kunštar, 2011; Wegst, 2006).

Modulus of elasticity, i.e. Young modulus, is one of several properties that have been assessed as music (resonance) wood quality, often presented in a form of specific modulus of elasticity, and is directly related to propagation of sound velocity (Bucur, 2006; Ono & Norimoto, 1984). Several studies additionally emphasize the significance of other parameters, like sound damping and radiation (Obataya et al., 2000; Ono & Norimoto, 1983, 1984), impact of frequency range on vibration damping (Spycher et al., 2008; Zorič et al., 2019) and acoustic anisotropy (Bremaud et al., 2011; Bucur, 2006; Bucur & Chivers, 1991). In order to achieve good acoustic properties of wood, the need for good cellular organization and homogeneity is often emphasized at the wood anatomical level (Bremaud et al., 2011; Brémaud et al., 2012; Obataya et al., 2000). Less is known about the impact of cellular organisation on vibration damping and internal friction of wood ( $\tan \delta$ ). It is not clear, if the relationships are similar for softwoods and hardwoods, with either diffuse- or ring-porous structure. Studies report in this respect impact of percentage of parenchyma rays on vibration damping in radial wood direction (Yano & Yamada, 1985) and decreasing of transverse damping ratio with wood increasing density, indicating some effect of porosity (Aoki & Yamada, 1972).

In the maple wood with fiddleback figure, however, research on the direct impact of wavy growth on its physical, mechanical and acoustic properties is not extensive, where there are still many open questions

(Alkadri et al., 2018; Kudela & Kunštar, 2011). It is not clear whether this maple species is required thanks of its specific wood texture and visual preferences (Buksnowitz et al., 2007; Carlier et al., 2014) or better features that the wood of the sycamore maple without wavy grain.

## MATERIAL AND METHODS

At the sawn timber warehouse of a commercial supplier, we sampled 12 radially oriented sycamore maple boards, 40 mm thick and 4000 mm long. Half of the boards contained parts with some wavy grained wood, having typical fiddleback figure (FB) in radial as well as tangential wood plane. Oriented test specimens ( $30 \times 30 \times 550 \text{ mm}^3$ ), two per board, were made at locations closer to the bark, and conditioned in a normal climate ( $20 \text{ }^\circ\text{C} / 65 \text{ \% RH}$ ). The end parts of the test specimens, 50 mm long, were used to determine the growth ring width (surface scanning of cross section, 1200 DPI, ImageJ) and dimensional stability of wood and for light microscopy (Leica SM2000R, Nikon Eclipse 80i). The dimensional stability of wood was analysed on small biscuits,  $30 \times 30 \times 5 \text{ mm}$ , by determination of differential swelling in radial ( $q_R [\%/ \%]$ ) and tangential direction ( $q_T [\%/ \%]$ ), after conditioning in dry ( $20 \text{ }^\circ\text{C} / 35 \text{ \% RH}$ ) and humid climate ( $20 \text{ }^\circ\text{C} / 33 \text{ \% RH}$ ), according to DIN 52 184 (Fig. 1a).

Specimens for mechanical tests ( $30 \times 30 \times 500 \text{ mm}^3$ ) were first examined by ultrasound (Proceq Pundit PL-200,  $f = 54 \text{ kHz}$ ), to determine time of flight and ultrasound velocity in three anatomical wood directions ( $v_L$  - longitudinal,  $v_R$  - radial,  $v_T$  - tangential; Fig. 1b). The modulus of elasticity ( $E'_L$ ) and vibration damping ( $\tan \delta_L$ ) were additionally determined by free excitation and the frequency response in the transverse direction of the oriented specimens (NI-9234, PCB130D20, NI LabVIEW; Fig. 1c) (Straže et al., 2015). Static mechanical testing was finally used to determine static modulus of elasticity and bending strength of wood (ZwickZ100, TestExpert3.0, EN 408; Fig. 1d).

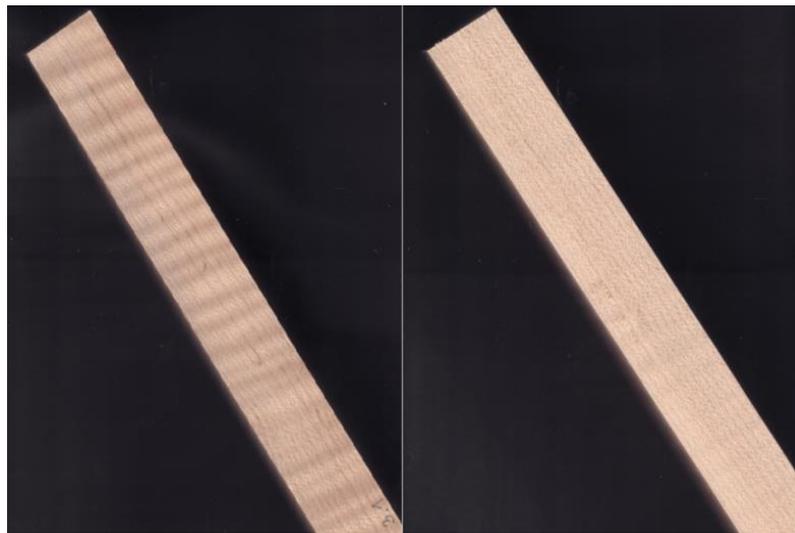


**Figure 15: Determination of dimensional stability (a), ultrasound velocity and moduli of elasticity (b), vibration damping (c) and static bending strength (d) of straight grained and wavy sycamore maple**

## RESULTS AND DISCUSSION

### *Structural and anatomical properties*

Growth ring characteristics of two tested categories of maple wood in the study were similar. The average growth ring width of straight grained maple (C) was 2.68 mm (Coef. of variation, CV = 10.82 %), comparable to the mean growth ring width of 2.79 mm at wavy maple (FB, CV = 17.92 %). In both cases, we did not confirm the characteristic relationship between the growth ring width and the density of the wood, which was expected for diffuse porous hardwoods (Alkadri et al., 2018; Diaconu et al., 2016; Emmerich et al., 2019). The mean wavelength of the wavy grain at maple was 8.97 mm, representing 11 waves per length of 100 mm, and the values varied considerably (CV = 13.67 %, Fig. 2). The value is slightly lower than reported by some previous studies (Alkadri et al., 2018; Krajnc et al., 2015).



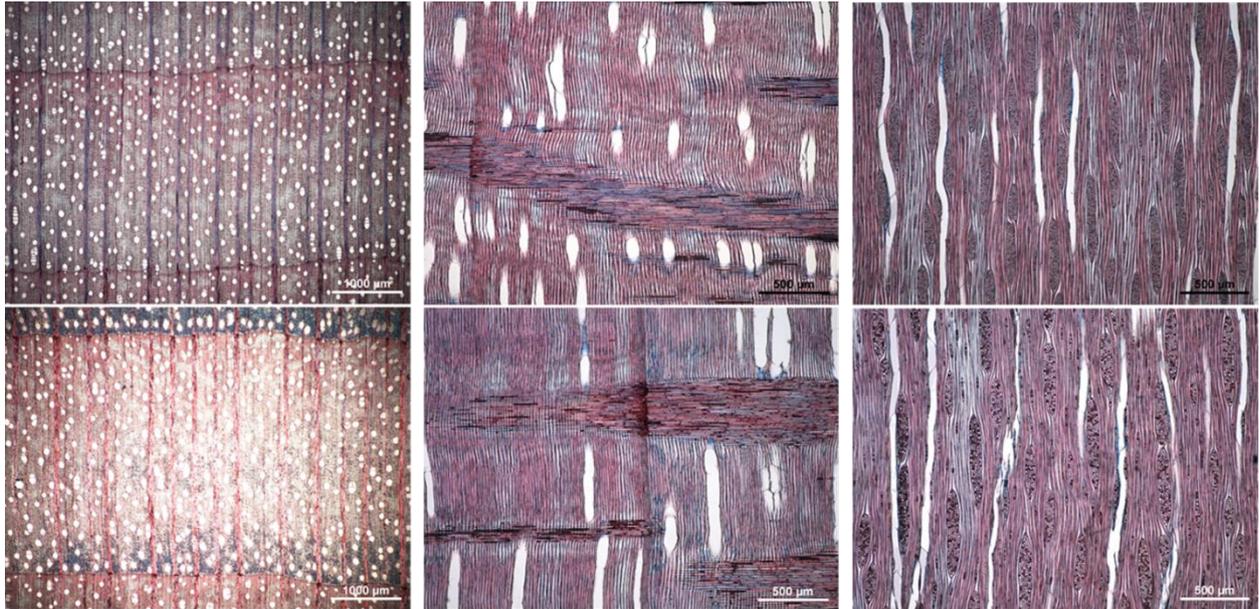
**Figure 16: Macroscopic radial-longitudinal plane of wavy- (left) and straight grained (right) sycamore maple**

In the qualitative analysis of the anatomical properties of straight grained maple and maple with fiddleback figure, no significant differences were detected. In our case, we measured only a slightly larger maximum pore diameter in maple with fiddleback figure ( $D_{\max, C} = 110 \mu\text{m}$ ,  $D_{\max, FB} = 135 \mu\text{m}$ ; Fig. 3). Similarly to previous studies, the waviness of wood grain was not detected at any of microscopic magnification (Alkadri et al., 2018), but contrary, this research did not find more tightly packed parenchyma rays in wavy maple.

The wavy growth of maple wood at the macroscopic level did not affect its dimensional stability (Table 1). We confirmed the good dimensional stability of both straight grained- and wavy maple in the radial and tangential directions. Due to the slightly greater differential swelling in the tangential direction, in the plane of the wavy growth, we confirmed slightly higher transverse shrinkage anisotropy in the maple with fiddleback figure.

**Table 1: Mean values of differential swelling in radial- ( $q_R$ ) and tangential direction ( $q_T$ ) and transverse shrinkage anisotropy ( $q_T/q_R$ ) of straight grained (control) and wavy sycamore maple (CV% - coef. of variation)**

		$q_R$ [%/%]	$q_T$ [%/%]	$q_T/q_R$ [ ]
Control	Mean	0.18	0.32	1.85
	CV%	16.7	12.5	16.8
Wavy	Mean	0.17	0.34	1.99
	CV%	17.6	2.9	18.6



**Figure 17: Transverse (left), radial (middle) and tangential section of straight grained (above) and wavy sycamore maple**

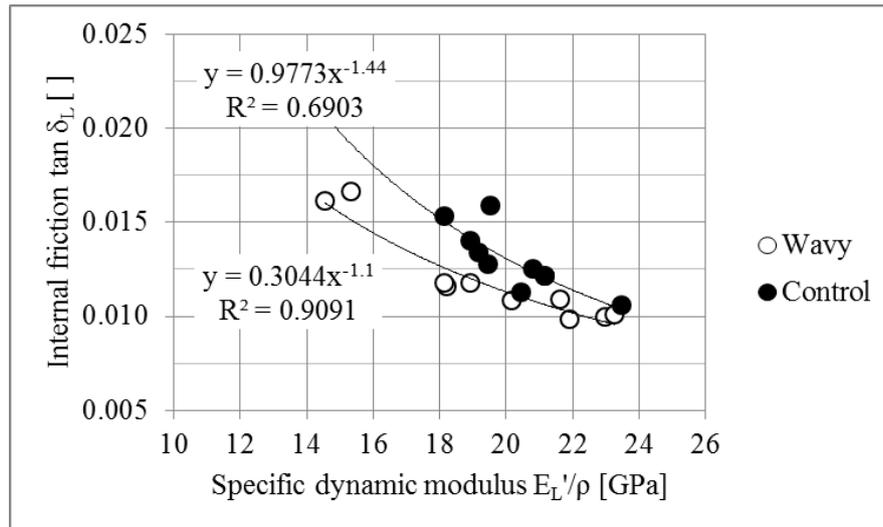
***Mechanical and acoustic properties***

The density of the studied straight grained and wavy sycamore maple was practically identical (Table 2), found in some previous studies (Alkadri et al., 2018), however contrary to some other findings (Bucur, 2006; Kudela & Kunštar, 2011). Negligible differences were also detected in the speed of ultrasound in the longitudinal ( $v_L$ ) and radial directions ( $v_R$ ), but not in the tangential direction ( $v_T$ ). In the latter, the speed of ultrasound in maple with fiddleback figure was significantly lower than in straight grained maple ( $v_{T,c} = 1312$  m/s,  $v_{T,FB} = 1165$  m/s). Using the known equation ( $E_i = \rho \cdot v_i^2$ ), and taking into account the comparable density of both categories of wood, the same relations apply to both in the case of modulus of elasticity in individual anatomical directions (Table 2). From the point of view of mechanical anisotropy, it was higher in wavy maple, which is due to the greater difference in stiffness longitudinally and transversely (tangentially) ( $E_L/E_{T(FB)} = 4.14$ ;  $E_R/E_{T(C)} = 3.71$ ) to the grain, as well as in radial and tangential direction ( $E_R/E_{T(FB)} = 1.62$ ;  $E_R/E_{T(C)} = 1.45$ ).

**Table 2: Mean values of wood density, ultrasound velocity in longitudinal- ( $v_L$ ), radial- ( $v_R$ ) and tangential-direction ( $v_T$ ) and equivalent moduli of elasticity ( $E_L$ ,  $E_R$  and  $E_T$ ) of straight grained (control) and wavy sycamore maple (CV% - coef. of variation)**

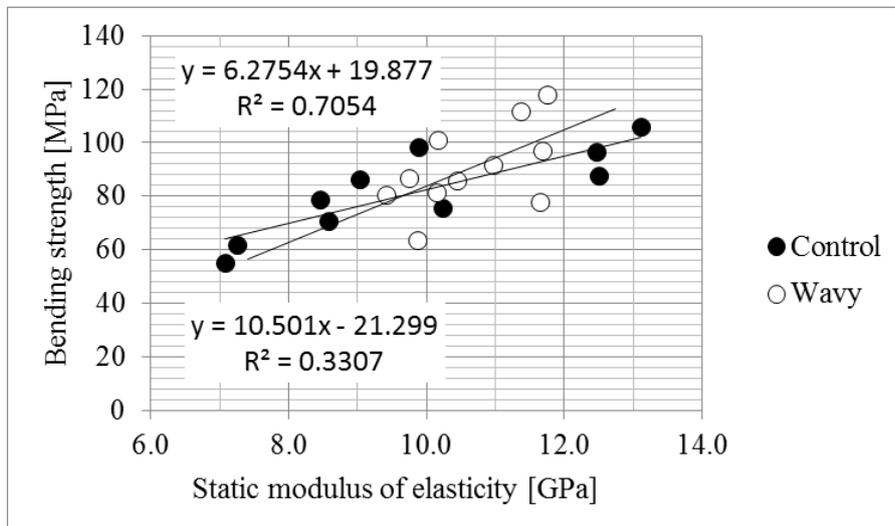
		$\rho$ [kg/m <sup>3</sup> ]	$v_L$ [m/s]	$v_R$ [m/s]	$v_T$ [m/s]	$E_L$ [GPa]	$E_R$ [GPa]	$E_T$ [GPa]
Control	Mean	626	4784	1862	1312	14.56	2.18	1.08
	CV%	0.95	3.57	3.98	7.64	6.39	8.19	14.30
Wavy	Mean	627	4849	1848	1165	14.80	2.16	0.86
	CV%	4.35	7.13	6.36	14.89	17.63	16.14	27.69

Pleasant, i.e. a sufficiently small damping of mechanical vibration is, in addition to adequate stiffness, one of the basic criteria when choosing wood for making musical instruments (Bucur, 2006). Both categories of maple wood proved to have a small damping of sound along the fibres ( $\tan \delta_{L(FB)} = 0.012$ , CV = 20.5 %;  $\tan \delta_{L(C)} = 0.013$ , CV = 4.0 %). The relationship between  $\tan \delta_L$  and  $E_L$  (determined by flexural frequency response) of sycamore maple follows a trend similar to the relationship established on numerous species (Bremaud et al., 2011; Ono & Norimoto, 1983; Straže et al., 2015) and maple as well (Alkadri et al., 2018) (Fig. 4). Slightly lower damping with respect to the specific modulus of elasticity was found in wavy maple, however not significantly different.



**Figure 18:** Relation between the damping coefficient (or internal friction  $\tan \delta_L$ ) and specific dynamic modulus of elasticity for straight grained (●) and wavy (○) sycamore maple

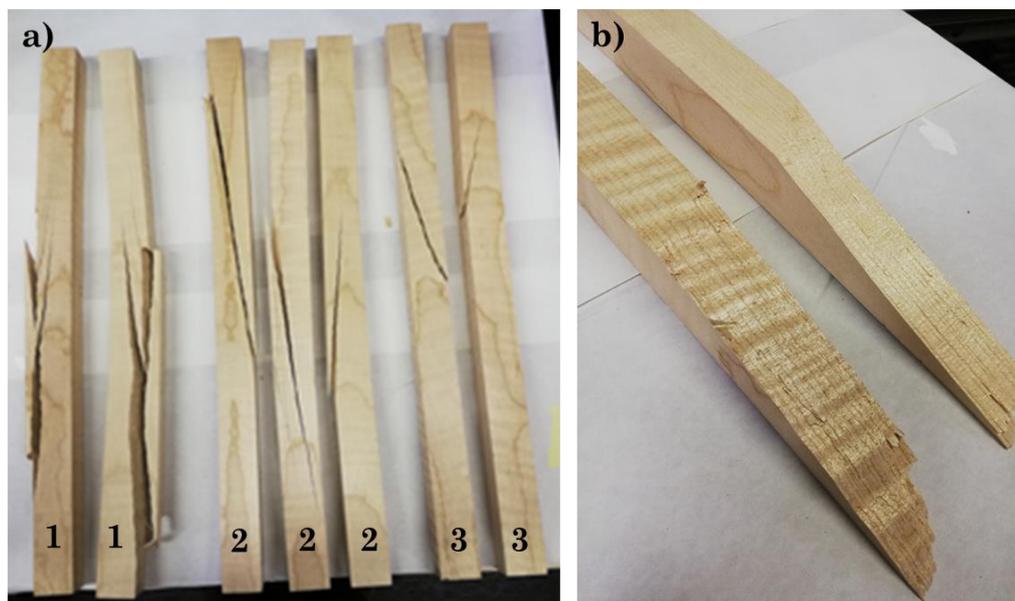
In static tests, the average modulus of elasticity of 9.36 GPa (CV = 22.2 %) was determined for straight grained maple, which was slightly higher, but not significantly different for maple with fiddleback figure ( $E_{S(FB)} = 10.28$  GPa). As expected, the values of the static modulus of elasticity of both categories of maple were lower than the dynamic modulus of elasticity obtained in ultrasonic testing and at frequency response experiments. The reasons are to be found in the shear effects in static bending testing (Boström, 1999), as well as in the influence of characteristic time and frequency in dynamic tests (Divos & Tanaka, 2005; Haines et al., 1996; Ouis, 2002). Static tests also confirmed the relationship between the flexural strength of both categories of test specimens and their modulus of elasticity, however more reliable at straight grained maple (Fig. 5). The average bending strength of straight grained maple was 89.88 MPa (CV = 19.0 %), and for wavy maple it was even higher, but insignificantly, namely 99.08 MPa (CV = 14.6 %).



**Figure 19:** Relation between the bending strength and static modulus of elasticity for straight grained (●) and wavy (○) sycamore maple

Majority of the specimens of both maple categories showed a brittle failure mode at ultimate bending stress. This mode of fracture can be partly explained by the presence of a mild to increased slope of grain, which was detected in a large number of specimens in both categories of maple wood (Fig. 6a). Ductile failure was detected as well in both categories of maple, with typical failure in tension zone and secondary shear

zone of specimens. In case of present strong deviation of slope of grain, wavy fracture planes revealing wavy growth pattern, were detected at maple specimens with fiddleback figure (Fig. 6b).



*Figure 20: a) Typical fracture patterns of the bending specimens, present at both maple categories: 1-failure in tension zone with secondary shear failure, 2-failure due to mild slope of grain, 3-failure due to increased slope of grain, b) Fracture section of wavy and straight grained maple*

## CONCLUSIONS

The present study confirmed only a few minor differences in the physical, mechanical, and acoustic properties of straight grained sycamore maple and maple with present fiddleback figure. Generally, no significant differences between these two categories of maple wood were found microscopically. Both categories of sycamore maple have similar density, dimensional stability and stiffness along the grain and in the radial direction. The maple wood with fiddleback figure has only slightly lower stiffness in the tangential direction, which increases its mechanical anisotropy. The density and bending strength of straight grained and wavy maple are similar as well, the same is true also for vibration damping along the grain, which is low and in favour to take advantage of both categories of sycamore maple in acoustics. Nevertheless, wavy maple, due to its specific texture, has an advantage in the use for making musical instruments.

## ACKNOWLEDGEMENT

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## Long-term effect on the equilibrium moisture content in thermally modified *Quercus cerris* L. Wood

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**Keywords:** Turkey oak, moisture content, long term effect, sapwood, heartwood

### ABSTRACT

This work aims to observe the combined effect of steaming and temperature on the equilibrium moisture content (EMC) of Turkey oak wood (*Quercus cerris* L.) and to check whether there are some changes or becoming stabilized during the years. Although several studies on the thermal modification to change wood properties have been investigated in short-term, there is no information regarding long-term effects, which are needed to understand the natural behaviour of a raw material such wood. In this study, Turkey oak wood has been subjected to five thermal treatments (please see Ferrari et al., 2013 for further details) and then stored under stable T and U conditions for ten years, at average values of 20°C e 63%, respectively. Samples were divided into heartwood and sapwood and results were compared to control. A total of 236 samples were monitored and finally soaked in water for 120 hours. Frequent mass monitoring of the samples in the selected period of the decade and the water uptake observation during the long term was essential to answer the two main research questions: How the wood behaves when stored for a long time in indoor and outdoor conditions? Does the sapwood and heartwood reaction are homogeneous or heterogeneous?

### INTRODUCTION

Wood utilization techniques are still related more to the application trend than to the technical culture that wood and wood-based materials would presume. Lack of wood characteristics and performances knowledge of designers can represent somehow the weak point of wood application. The wood modifications can be defined as an improvement of the intrinsic properties of this material, producing a new one with limited emissions, with full respect for the environment (Hill 2006).

Heat treatment of wood is widely studied, as early as Tiemann (1920) showed that by subjecting it to high temperatures occurred a decrease in the equilibrium moisture content, with a consequent decrease in dimensional shrinkage.

Several studies on the thermal modification to change wood properties have been investigated with focus on short-term period, but there is no detailed information regarding long-term effects. In the present work the investigation to determine the long-term reaction (ten years) of wood samples subjected to different thermal and steaming treatments was carried out to enhance the Turkey oak wood workability going beyond its current energy purposes.

### EXPERIMENTAL METHODS

The experimental in this research was carried out to assess the behaviour of the Turkey oak wood, previously subjected to various types of hydrothermal treatments (Fig. 2), in the long-term. The logs come from Mediterranean high forest oak located in the Apennine Mountains of Basilicata, south of Italy (Fig.1).

After sawing on 30 cm thick boards, specimens used for our purpose were extracted from the raw material naturally seasoned.

The data were taken on samples of size 20mm (tang) x 20mm (rad) x 40mm (axial) on which the various thermal treatments were carried out. More details are in Ferrari et al., (2013).



**Figure 1: Logs before the experimental**

Once selected they were separated into several groups, which were made up considering proportionality in sapwood, heartwood and mixed wood amount. Whenever a group of specimens has undergone thermal modification, its weight has been measured, so that any changes can be noted. A single group (the number 6), called "control", has not undergone any treatment, to make a final comparison with the various groups treated. Then each group was treated differently.

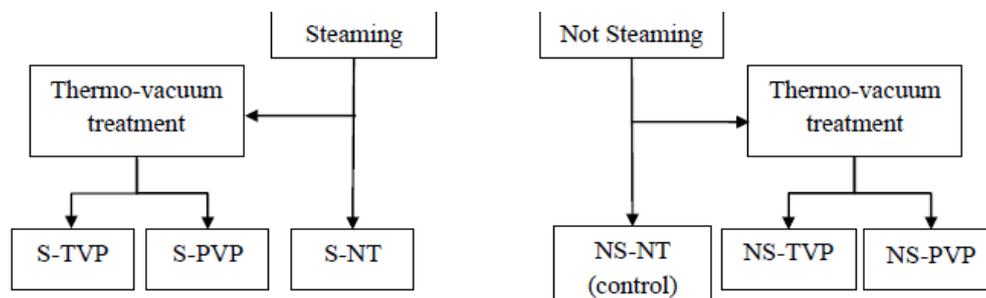
The wood samples were divided into 6 groups, of which 5 underwent treatments:

- group 1: S-PVP
- group 2: NS- PVP
- group 3: S-TVP
- group 4: NS-TVP
- group 5: S-NT
- group 6: NS-NT.

All the treatments lasted 3 hours at a temperature of 170 °C. After the treatments, the samples were placed in the climatic cell with constant temperature (20 °C) and relative humidity (65%), to gain the equilibrium moisture content.

The changes in weight due to the absorption of humidity were detected initially every day, then weekly and finally annually.

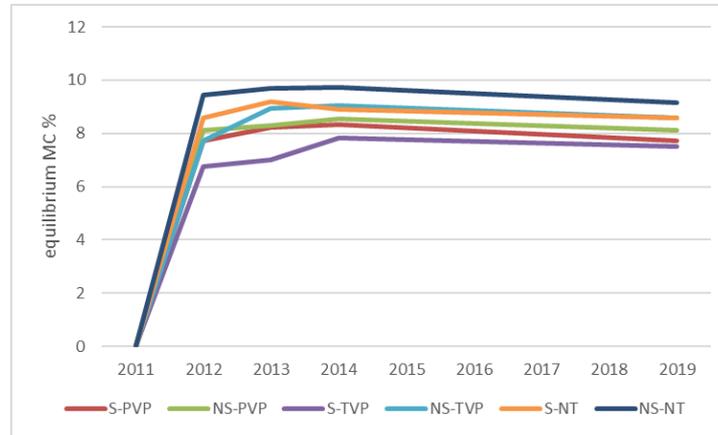
After ten years, the Turkey oak samples, after being analysed according to the different treatments, underwent a 5-day immersion process to assess the absorption capacity of the different types of treatment, distinguishing sapwood from heartwood.



**Figure 2: Treatment pattern. S=steamed; NS=not steamed; NT= no thermo-vacuum; PVP= thermo-vacuum with press-vacuum technology; TVP= thermo-vacuum with Termovuoto® technology**

## RESULTS AND DISCUSSION: HYGROSCOPICITY IN THE LONG PERIOD

As shown in the Graph. 1, in the indoor condition the different types of treatments have not led to substantial differences in hygroscopic absorption capacity, as they have all used the same temperature (170 °C), the untreated samples are the most sensitive to atmospheric humidity, followed by the steamed group.



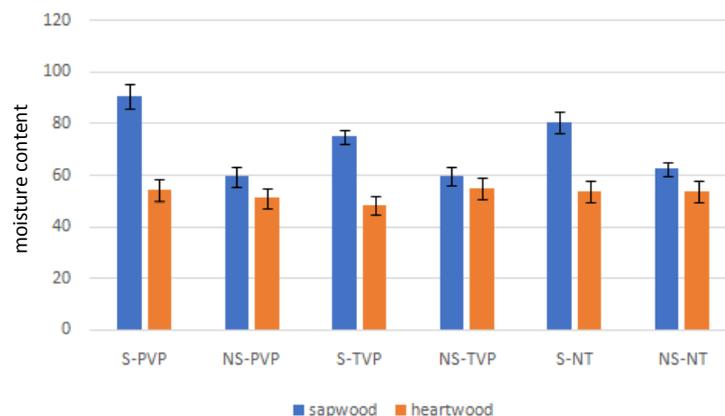
**Graph. 1:** % Turkey oak wood humidity at the various treatments over time.

At the up-mentioned treatments, the samples immersed into the water for 120 hours, have been evaluated according to the absorption capacity in a saturated environment. From the Graph. 2 can be noticed the sample groups that have undergone major changes are the vaporized ones. Therefore, in this case, they are not only the result of the reaction to temperature, but it can be stated that the steaming treatment in the autoclave at a temperature of 110 °C for 24 hours at 140kPa of pressure, has increased the water absorption capacity of wood. A possible explanation could be that this type of treatment modifies the microscopic structure, eliminating the tylosis present in the vessels, increasing empty spaces. This can be considered as an unfavourable factor for wood commercialization, mainly in outdoor condition with the presence of high humidity level, if it is intended to be used for industrial/construction purposes and not only as firewood. This phenomenon would increase the burning capacity of the wood, causing a decrease in its main characteristics, which is a slow release of heating energy due to fewer pores occupied by air.



**Graph 2:** moisture content of Turkey oak wood post immersion.

As for the sapwood and heartwood water absorption capacity, after thermal modification, there is a significant difference between the two types of wood, as shown in the Graph. 3.



**Graph. 3: absorption differentiation in sapwood-heartwood after the immersion.**

The treatments where the difference in water absorption capacity increases were those that have undergone steaming treatment.

## CONCLUSIONS

The steaming treatment previously carried out has significantly changed the water absorption capacity of the wood species, mostly after water immersion. Sapwood and heartwood respond differently, but since in the wood industry it is difficult to identify and differentiate those two types of wood, the steam treatment would be not recommended for outdoor applications and therefore, they would be exposed and completely defenceless from the weather.

The enhancement of this wood species could be not only added value for wood utilization in our territories, both on a local and national scale, considering it a species easy to cultivate and perfectly adaptable to the foothills of our country. In Basilicata (as widely spread in the rest of south-eastern Europe) the adult oak forests are  $\pm 83'625$  Ha (© SIAN.it). This would reduce significantly the imported timber amount from abroad, that would reflect in an increase in employment for local workers. By carrying out the necessary studies, the intended use of this wood could be changed by inserting it in the construction sector. The equilibrium moisture content in the long period is reached only with thermal modification application. Otherwise, the steaming treatment caused a dimensional instability. After immersion, the heartwood keeps the observed differences instead of sapwood, who increased its water absorption capacity. This can be a guideline for the outdoor application of this species. Based on the findings of this research, various parameters can be considered of considerable importance, who may affect the woodworking companies' decisions. Deeper studies are necessary to confirm the modified physical and technological properties found following thermo-hygrometric treatments.

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## Microwave modification and drying of Jarrah (*Eucalyptus marginata*) boards

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**Keywords:** Jarrah, lumber, microwave drying, microwave wood modification, specific cost, wood permeability.

### ABSTRACT

Very low permeability of Jarrah (*Eucalyptus marginata*) species causes problems during lumber drying. These include: very long drying times, drying defects, and large material losses after drying, high energy consumption and expensive drying processes. Microwave (MW) wood modification and MW drying can provide an increase in wood permeability and solve some hardwood drying problems.

Two 60 kW MW conveyor plants at frequencies 2.45 and 0.922 GHz were used for experiments. The study of MW Jarrah (29x87mm) board drying after pre-drying MW modification showed that it is possible to dry boards in the moisture content range from 70 to 10% during 147-159 hours (6 -7 days) using cycle method of MW power application. Such intensive MW drying does not bring a perceptible damage to the wood and provides low variability of moisture content in the board cross sections. The shorter drying times reduce associated capital, space, energy and labor costs whilst the reduction in drying defects can increase yields. Short time of MW drying provides opportunity to develop conveyor systems for hardwood sawn timber drying.

Economic assessment of MW Jarrah board drying showed that a specific drying cost ranges from US\$ 81 to \$137/m<sup>3</sup> at electricity prices from US\$0.06 to \$0.12/kWh. The electricity costs form the largest share in total specific drying costs - 41 to 65%. Jarrah boards are expensive product and MW drying costs \$ 81 - \$137/m<sup>3</sup> can be acceptable for industry taking into consideration savings provided by fast drying and wood loss reduction.

### INTRODUCTION

The timber industry has permanent problems associated with hardwood drying. These include very long drying times, drying defects, and large material losses after drying, high energy consumption and expensive drying processes. Jarrah (*Eucalyptus marginata*) species drying is very long process and requires special schedules to prevent drying defects. Jarrah commercial drying operations impose an extended period of slow air drying to reduce the incidence of drying defects (checking and collapse) prior to kiln drying. Microwave (MW) wood modification can solve some wood drying problems. MW pre-treatment increases the permeability of the green wood (Torgovnikov and Vinden 2009) overcoming the propensity of hardwoods to collapse during kiln drying and provides opportunities for fast kiln drying immediately after sawing. Also after MW pre-treatment the application of MW for lumber drying can provide fast drying and reduce drying defects (Harris et. al. 2008).

An experimental study was carried out to determine a feasibility and economic assessment of microwave drying of Jarrah boards after MW pre-drying treatment. The MW drying trial incorporated development of a schedule for board drying and subsequently provide data for processing and costing. The drying schedules needed to be able to dry the timber rapidly to produce a good quality product (Compare 2005). Objectives of the research work included experimental study of MW interaction with Jarrah sawn timber in the MW applicators; study of the effects of MW drying on wood quality; possibility of Jarrah boards conveyor drying; energy consumption for drying boards; and an economic assessment of sawn timber MW drying.

## MATERIAL AND METHODS

### Material

Twenty green Jarrah boards (Back-sawn and Q- sawn) measured 29x87x3000 mm were used to determine an appropriate microwave schedule and conduct the drying trial. The initial moisture content was determined by oven drying to be in the range of 65 to 78 % and average 69%. Oven dry density was in the range of 780 to 890 kg/m<sup>3</sup> and average 840 kg/m<sup>3</sup>.

### Experimental equipment

MW board drying process included two stages:

- Pre-drying treatment - wood structure modification by intensive MW conditioning to increase wood permeability using frequency 2.45 GHz,
- MW drying using frequency 0.922 GHz.

To increase wood permeability of the green boards, 2x30 kW MW roller conveyor plant (Fig.1) at frequency 2.45 GHz with 2 port applicator (cross section 115x115 mm) was used for pre-drying boards treatment. Radiator slot size was 20x86 mm. Electric field strength vector E in the radiators was orientated parallel to the wood grain. Scheme of applicator is shown in Fig. 1. Power 10 kW was applied from one generator from two sides via power divider (50 +50 %). Board speed in Applicator varied in range 12-20 mm/sec with power supplied to the timber from 7 to 10 kW required for wood modification.



Figure 1: MW conveyor plant for timber modification 2x30 kW (frequency 2.45 GHz) and board position in 2 port MW Applicator (115x115x350 mm)

A 60kW MW conveyor installation at frequency 0.922 GHz (Fig. 2) was utilized for timber drying. The installation included a microwave power supply, waveguides, tuner, microwave applicator, water load, conveyor feed system and dynamic air system for the removal of vapors from the applicator and the prevention of water condensation on the walls of the applicator.

Technical data for the MW conveyor installation:

- Microwave power output 10-60 kW
- Microwave frequency 0.922 GHz
- Max dimensions of timber 90x90x4500 mm
- Feed speed range 6-60 mm/sec
- Air heating power 13 kW
- Air temperature 20-150 oC

Tunnel of MW applicator had cross section 124x 248 mm. Electric field strength vector E orientation was perpendicular to the wood grain. The timber was located within the conveyor applicator with the long size of the lumber cross section in the horizontal position (Fig. 2).

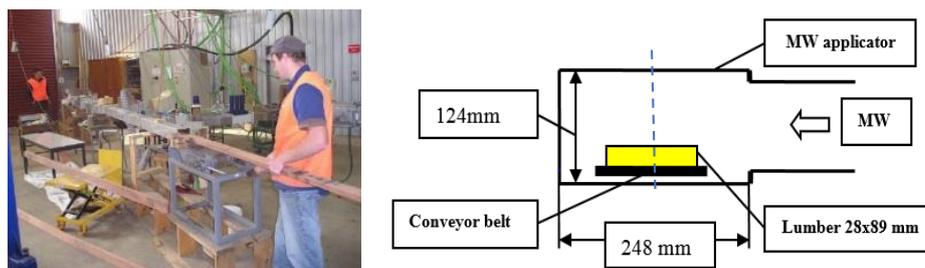


Figure 2: 60 kW microwave conveyor installation (frequency 0.922 GHz) for board drying and applicator scheme

## ***Experiment procedure***

### **Stage 1. Pre-drying MW wood modification for increasing permeability.**

In the beginning the pre-drying rational MW schedule was determined by measuring temperature distribution in board cross sections after board processing in 2 port Applicator of 2x30 kW MW plant (Fig. 1). Different MW power was applied to the boards at different timber speeds to get energy distribution in board cross section required for wood modification (100 °C during the short period of time) to increase the wood permeability. After tests the following pre-drying MW Schedule 1 was chosen:

- MW power 10 kW (2.45 GHz)
- Energy supplied 69 kWh/m<sup>3</sup>
- Board speed 16 mm/sec
- Air temperature 80-90 °C

### **Stage 2. Microwave conveyor drying.**

After pre-drying treatment the drying schedule was determined by preliminary drying boards in conveyor installation (Fig. 2) to prevent board overheating and cracking. After tests the following cycle drying Schedule 2 was chosen:

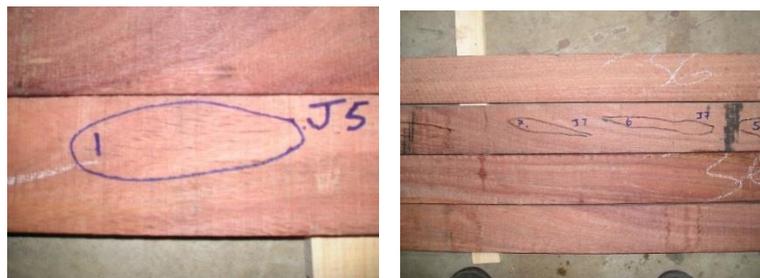
- MW applied power 4 kW (0.922 GHz)
- Conveyor speed 52 mm/sec
- Air temperature in conveyor tunnel 40 - 45 °C (at speed 0.5 -1 m/sec)
- Number of board passes via applicator in 1 cycle 3
- Time of 1 pass 58 sec
- Drying procedure:
  - Cycle 1
    - a) 3 passes ( MW heating)
    - b) cooling (drying) 1 hour at room temperature (16-17 °C)
  - Cycle 2
    - a) 3 passes ( MW heating)
    - b) cooling (drying) 1 hour at room temperature (16-17 °C)

and so on.

Procedure for MW pre-drying treatment and MW drying of the boards:

1. Surface check assessment and marking on board surfaces
  2. MW pre-drying treatment according Schedule 1
  3. Surface check assessment and marking (Fig. 3)
  4. Weighting after MW pre-treatment
  5. MW drying according Schedule 2 (3 runs)
  7. Weighting after 3 passes via MW applicator
  8. Cooling - drying during 1 hour in the room at temperature 16-17 °C
  9. Weighting after 1hour cooling-drying
  10. MW drying according Schedule 2 (3 runs)
  11. Weighting after 3 passes via MW applicator
  12. Cooling - drying during 1 hour in the room at temperature 16-17 °C
  13. Weighting after 1hour cooling-drying
- and so on.

The drying process proceeded until the board moisture content 8-10%.



***Figure 3: Surface check assessment and marking on the board surfaces***

To determine moisture content on the width of the board after drying three pieces of 10 mm thickness were cut from both sides and the middle of every board.

## RESULTS

Boards were MW modified according Schedule 1 and MW dried according Schedule 2. After MW modification the boards lost about 1.6 % moisture content. Drying cycle had 72 min: 12 min MW heating and 60 min cooling-drying. Boards had 134 -138 drying cycles. Drying curves are shown in Fig. 6. Drying of the boards with initial moisture content 66-72% up to 12% takes 123-131 hours (average 127 h) and up to 10% - 147- 159 hours (average 153 h). Board drying curves are shown in Fig. 4.

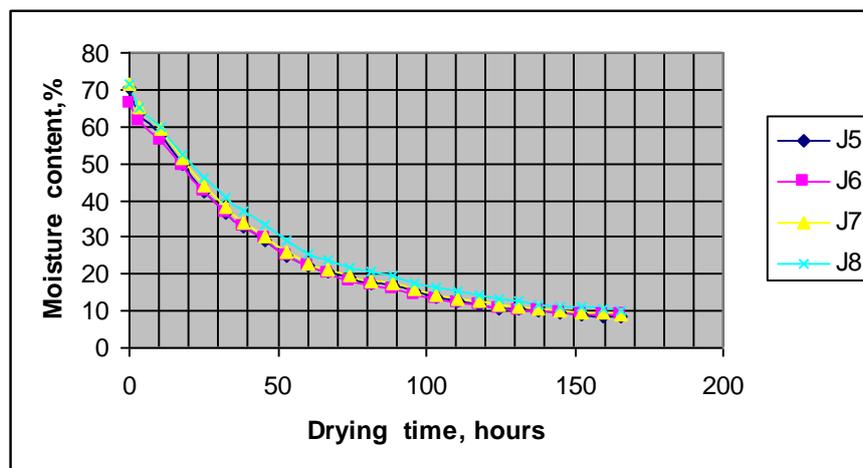


Figure 4: MW drying curves of Jarrah 29x87 mm board numbers J5, 6, 7, 8.

After MW modification visible surface checks appeared in some places on two (Back-sawn) boards (see Fig. 3). After drying all the surface checks closed but some of them was possible to see on cross section. Q-sawn boards had no checks. The shapes of the board cross sections after drying are shown in Fig. 5.



Figure 5: Jarrah board cross sections after MW drying

The moisture content difference in the board cross sections after drying was in the range 1-2 %. On the final stage of drying after 3 passes of MW heating the temperature inside the boards reached 100-108°C but it did not bring any visible damage to the wood.

In the drying process the energy is spent for timber heating and water evaporation. MW energy consumption for pre-drying treatment is 69 kWh/m<sup>3</sup>. MW energy consumption for Jarrah lumber drying in the moisture content range from 70 to 10% is about 490 kWh/m<sup>3</sup>. Total MW energy consumption is 560 kWh/m<sup>3</sup> and electric energy consumption is about 660 kWh/m<sup>3</sup>. It does not include the energy required for heating of circulating air.

Experiments with Jarrah board MW drying showed that it is possible to dry boards in the MC range from 70 to 10% during 147-159 hours (6-7 days) using cycle method of MW power application. Such intensive MW drying does not bring a perceptible damage to the wood. The 1-2 % moisture content difference in the board cross sections after drying is reasonable. On final stages of drying after 3 passes of MW heating the temperature inside the boards reached 100-108°C, but it did not bring any visible damage to the wood. MW timber modification and drying overcomes the propensity of the collapse and reduces the losses of timber due to long storage times. Traditional Jarrah sawn timber drying requires the lengthy 3-4 months air drying applied before kiln drying during 2-3 weeks (Australian Timber Seasoning Manual 1997). MW conveyor drying allows to reduce drying time significantly up to 6-7 days (14-20 times). This study does not allow to estimate all aspects and advantages of fast MW conveyor drying and further research required.

### PRELIMINARY ECONOMIC ASSESSMENT

The economic assessment for microwave Jarrah timber drying is given based on the following conditions: MW cross conveyor plant for drying Jarrah boards from MC 70 to 10% with MW power 500 kW (frequency 0.922 GHz); plant outputs: 4,000 m<sup>3</sup>/year at 4,000 working hours per year (2 shifts per day) and 6,000 m<sup>3</sup>/year at 6,000 working hours per year (3 shifts per day); MW plant cost - US\$ 950,000; MW plant works automatic; electric energy consumption 660 kWh/m<sup>3</sup>; electricity cost range US\$0.06/kWh to \$0.12/kWh; depreciation rate 17%.

Calculated specific costs include costs associated with capital, maintenance, magnetron replacement, and electricity costs. These costs do not include cost of building, mechanical installation, electrical connections, on costs (overheads) and taxes.

Fig. 6 shows MW lumber drying specific costs depending on the plant output and electricity cost in the range US\$0.06/kWh to \$0.12/kWh at three working shifts per day.

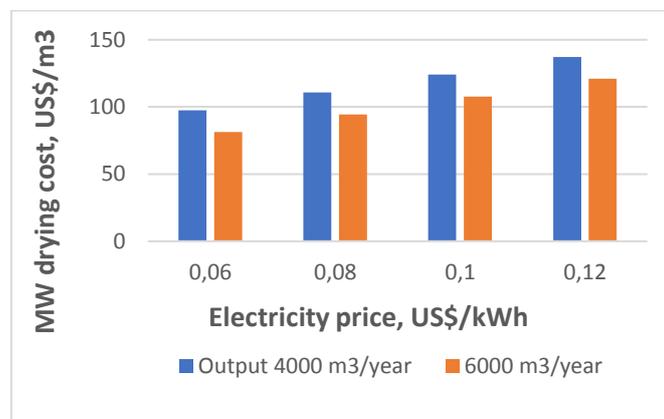
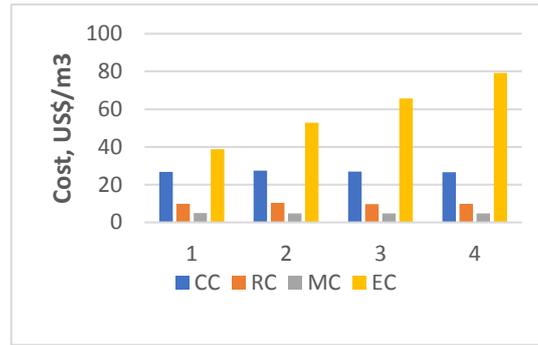


Figure 6: MW drying specific costs depending on plant output and electricity costs

Specific drying cost ranges from US\$98 to \$137/m<sup>3</sup> at 2 shifts per day and electricity costs from \$0.06 to \$0.12/kWh, and from \$81 to \$121/m<sup>3</sup> at 3 shifts per day. Drying plant output increase from 4,000 m<sup>3</sup> per year to 6,000 m<sup>3</sup>/y leads to specific cost reduction by 13-20%.

Fig.7 displays specific cost components: capital cost, magnetron replacement, maintenance, and electricity cost for plant output 6,000 m<sup>3</sup>/y at 3 shifts per day and electricity price from US\$0.06/kWh to \$0.12/kWh. The electricity costs in the price range \$0.06 - \$0.12/kWh form the largest share in total specific drying costs - 41 to 65%, capital costs form 22 to 41% share, and magnetron replacement costs form 8 to 12% share.



**Figure 7: Specific cost components: CC- capital cost, RC- magnetron replacement cost, MC – maintenance cost, EC- electricity cost. Plant output - 6,000 m<sup>3</sup>/y at 3 shifts per day and electricity price: 1 - US\$0.06/kWh, 2 - \$0.08/kWh, 3 - \$0.10kWh, 4 - \$0.012/kWh.**

Jarrah boards are expensive product with price US\$2200 to \$2600/m<sup>3</sup> and MW drying costs \$ 98 - \$137/m<sup>3</sup> can be acceptable for industry taking into consideration savings provided by fast drying.

## CONCLUSIONS

Experiments with MW Jarrah (29x87mm) board drying with pre-drying MW modification showed that it is possible to dry boards in the moisture content range from 70 to 10% during 147-159 hours (6-7 days) using cycle method of MW power application. Such intensive MW drying does not bring a perceptible damage to the wood and provides low variability (1-2%) of moisture content in the board cross sections. Electric energy consumption for drying Jarrah boards down from 70 to 10-12 % moisture content is around 660 kWh/m<sup>3</sup>.

The specific MW drying cost ranges from US\$ 98 to \$137/m<sup>3</sup> at two shifts per day and electricity prices from \$0.06 to \$0.12/kWh, and from US\$81 to \$121/m<sup>3</sup> at three shifts per day. The electricity costs form the largest share in total specific drying costs - 41 to 65%, capital costs form 22 to 41% share, and magnetron replacement costs - 8 to 12% share.

Jarrah boards are expensive product with price US\$2200 to \$2600/m<sup>3</sup> and MW drying costs \$98 - \$137/m<sup>3</sup> can be acceptable for industry taking into consideration savings provided by fast drying and wood loss reduction.

Short time of MW drying provides opportunity to develop conveyor systems for hardwood sawn timber drying. This study does not allow to estimate all aspects and advantages of MW conveyor drying and further research required.

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## Deciduous trees in architecture

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**Keywords:** architecture, interior design, design, organic, ecology, folkish

### ABSTRACT

Deciduous trees can be found in architecture in so many ways that we must talk about its curiosity, importance and the fact itself: in how many opportunities exists we can use these trees – as an instrument, as a tool for use: for design/for structural purposes etc.

This paper will focus on deciduous trees as a material which we can use for so different ways:

- traditional folkish architecture (gate, beam etc.)
- traditional folklore (chest, furniture, instrument etc.)
- contemporary architecture (showing the material itself, reusing waste, interior design, street furniture etc.)

In the traditional folkish architecture section, the paper will discuss black locust and oak as a hardwood material how it was used by Hungarian peasants. This part would like to point on how and why people could choose between species for an explicit duty.

In fact, traditional folklore is part of the folkish part – although it does not especially belong to architecture thus this will be discussed in another section. Special chests, chairs will be shown using traditional painting technique (technique of Harta).

Contemporary architecture divided in two parts: deciduous trees as building material used in architecture and in interior design.

### INTRODUCTION

The ideology and attitude of today's architecture wants architects to rethink building materials which were 'forgotten', use more 'green' resources and reuse the existing ones to minimize the energy needs while producing them. Nowadays (global crises and sudden population growth) we have similar problems as we had earlier (world wars, natural disasters): the lack of (natural) sources.

Centuries ago, it was obvious to build a simple house from trees and stones – although nowadays we cannot afford this: our houses are built from prefabricated, source (material) minimalized solutions.

We have to rethink our goals and possibilities. I think today's architects biggest mission is to help nature (full habitation) and to equalize the differences between nature and mankind.

Based on the facts mentioned above we will concern on some examples brought from the past and on those ones we have today to note: deciduous trees' importance and the wide range of solutions we can reuse waste materials to minimize them.

In this paper I would like to point to the fact: about more than a century ago people had a balanced life living with nature: they exactly knew which type of hardwood or softwood is capable for each function. Economical thinking was not an optional opportunity: the standard thinking itself was economical.

Through several examples we can see how different solutions could be made using hardwood.

With this remembering I would like to draw attention to nowadays' trends: it should not be a choosable option but a followed one.

## TRADITIONAL FOLKISH ARCHITECTURE

While centuries ago hardwood was an optional and clear option to solve everyday's needs – today we try to ransom or absolutely minimize hardwood as a material. We must pay attention to the usability of beech, oak, black locust and several kind of fruit trees (cherry, peach etc.) how great materials they were in folkish architecture. People knew which specie to use to satisfy their needs.

### ***Black locust (*Robinia pseudoacacia*)***

It was acclimatized in the 18<sup>th</sup> century in Hungary in the need of binding the drift sand (Wikipedia 2019). The Great Lowlands of Hungary was an ideal place for black locust: high hours of sunny days and vital soil. People needed a specie which can tolerate high temperature, dry weeks. Black locust was this (Wikipedia 2020a).

The folkish people had the talent to use the resources surrounded by. Black locust was that kind of tree to the Hungarian lowland people (lack of trees) which could be used as a strong hardwood beam above windows. Properties of this tree could not made it to support as a full length wooden joist, but a shorter one. The short length and inner tension did not let use it to long spans – although long spans did not need at all situations. Until nowadays we can find these support beams (Fig.1) in old peasant's houses and in farmhouses, too – black locust was a popular choice before inventing reinforced concrete and before industrial steel manufacturing.

This tree was also a great option to make handtools.

It is also an important fact, that black locust is also a decent tree producing honey. The flower of the tree can be used for treating coughing symptoms and for heartburn, too.



*Figure 1: The surface of the support beam is roughened in the interest of providing ideal surface to plaster (Pinterest 2020a)*

### ***Oak (Quercus spp.)***

The specie (oak) acclimatized to its habitat: for the effect of the ice age and the wandering of the continents (Wikipedia 2020b). Thus several species came alive and evolved several properties. The following example will show that the folkish people (Wikipedia 2020c), the “Székely”s (Hungarian ethnic group located in the southeast part of historical Hungary, today it belongs to Romania) discovered these advantageous properties of oaks: they created the “Székely kapu”s (gate of Székely).

These gates are made of (mostly) oak (Fig. 2) which are richly decorated with handmade carvings (symbols, words etc.). Essentially it is 2 several sized gates pieced together (Wikipedia 2020d) and covered with roofing (for pigeons).

The small (Fig. 3) gate and the big gate clamped together with a long beam (on the top of the three wooden pillars). The bigger gate created for hay carts to fit through, while the smaller one was for pedestrian traffic. The area made from the differences come from the size differences makes a great opportunity to decorate the gate. The exact ratio is established for width and length (approximately the big gate is two times bigger than the small one).

The most ideal oak specie is oak (*Quercus robur* and *Quercus petraea*) because of its high content of tannins. Due to this fact these species are especially a good choice against wood destroying fungi.

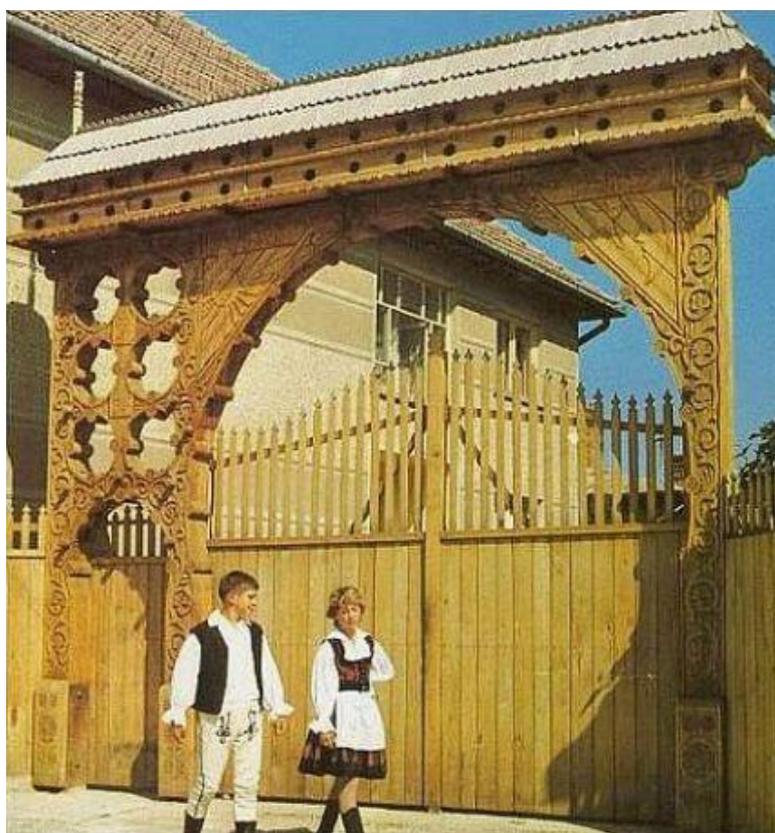
The exact tree is chosen before logging: the logged-out hardwood must be dried at least for a year.

The first step is to make the rectangular shaped (in cross section) pillars in half-dried hardwood state. These three pillars clamped together with a beam which supports them and the roof structure, too.

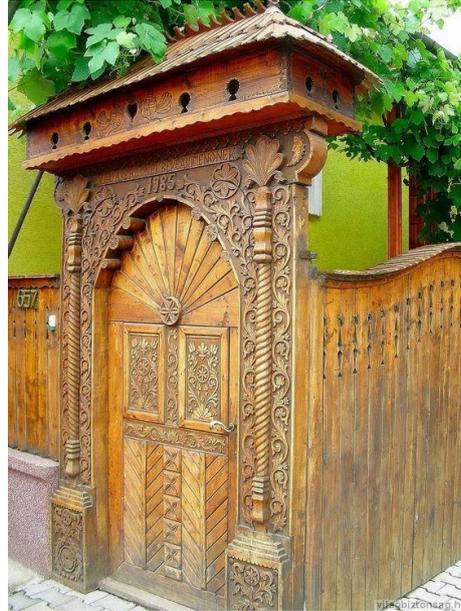
This is the main frame of the gate. With knitted wooden joints the other parts of the gate are made: brackets and rafters etc. Final step is to fill the area with wood above the small gate and to make the carvings and paintings. The area above the small gate called “crown” – which is the main place for decorating (Fig. 4).

This place is usually enriched with ornamentic symbols: tulips, birds, roses and with citations, names.

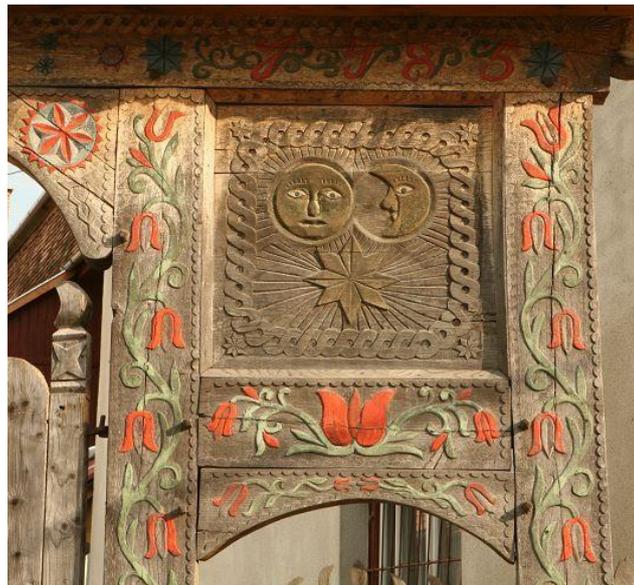
Each symbol has its meaning (for example the Sun is for blessing, and for man, Moon is for woman, tulips are for femininity etc.).



**Figure 2: Székely kapu (Székely’s gate), note the traditional (folkish) costumes (Pinterest 2020b)**



*Figure 3: richly decorated passenger gate, note this different symbols (Pinterest 2020c)*



*Figure 4: upper part of the small gate: the 'crown'(Pinterest 2020d)*

## TRADITIONAL FOLKLORE

In this section I would like to introduce a traditional Hungarian chest (Fig.5) (“szuszék láda”), a traditional folkish chair (Fig.6) painted with a more than 200 years old technique (technique of Harta) and my own made traditional Hungarian zither (Fig.7) spreaded in the lowlands of Hungary.

### ***“Szuszék láda” (chest made by joints with carpentry work)***

Hardwood splitted by axe where the joints are made with carpentry connections. It is specific that the plank fields are formed and connected between four corner pillars. The lid is usually shaped rounded where the rotation point granted at the back side by a wooden joint. Originally this type of chest designed to store clothes, serve as a coffin and later to become a cereal holder. Usually decorated with wooden runic symbols, sometimes it is painted (Fig.5). The name used (“szuszék”) by Hungarians spreaded at the northern and eastern parts of Hungary. The original chest was made of beech without glue.



*Figure 5: "Szuszék láda" decorated with runic symbols, © Gyenes Tamás (Gyenes 2020)*

***A traditional folkish chair decorated with painting technique of Harta***

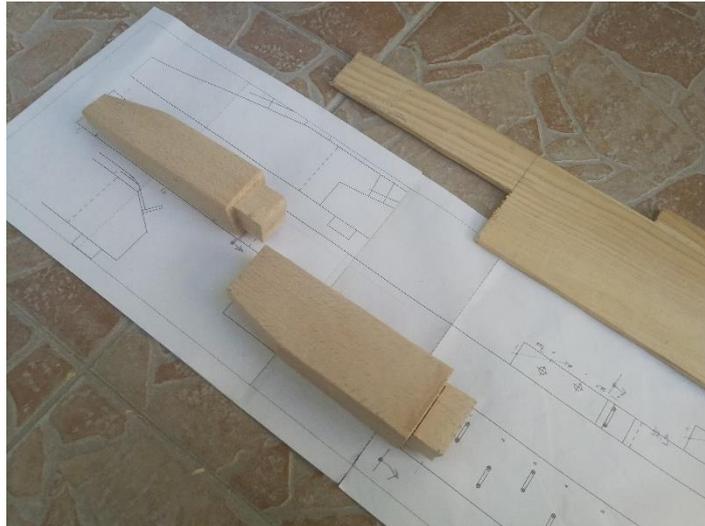
Harta is a municipality in Hungary, located in the middle of the country at the bank on the river Danube. Originally the painting technique of Harta was born more than 200 years ago and still alive. The main colour has always been blue. It is an often-used symbol: the bouquet (Fig.6). These kind of chairs are usually made of beech and oak.



*Figure 6: traditional folkish chair decorated with the painting technique of Harta (Schneider 2020)*

### ***A traditional Hungarian zither***

Zither is a stringed instrument layed on a table while playing on it. Although there are several types known, the simplest one is the prism formed (Wikipedia 2020e). The instrument is rectangular shaped, built with a hardwood flat area where the musician can hold down the strings to play music. This part is usually made of hardwood in the interest of resisting scratches more than other type of softwoods (Fig.7). Those types which are known that are made at the area of the Great Lowlands of Hungary named “Alföldi citera” (zither of the Great Lowlands). This type of instrument is a Hungarian value due to its style and history. The head and the bottom parts are made of hardwood because these parts are the most used ones while tensioning the strings and needs to resist these forces. The body is made of pine.



***Figure 7: The head parts of the zither made from beech (Fagus), while the body made from spruce (Picea)***

## **CONTEMPORARY ARCHITECTURE**

Today we can use the waste from wood industry in architecture, too: the end of the wood can be used as an interior pavement for example (Fig.8).



***Figure 8: Oak parquet flooring: great choice for using waste material (ildare 2020)***

Using several different waste parts of deciduous trees (smaller pieces, branches, barks etc.) we can build usable products, too (Fig.9).



*Figure 9: using waste parts of the tree to build a new product (Ana 2011)*

## CONCLUSIONS

It has been more than a century we use plastic to ransom basic materials as much as possible. Calculating the energy, we need to produce plastic and after usage the energy to reuse them compared to hardwood production we can consider through numbers how economical is to use plastic or hardwood.

Illustrated through traditional folkish solutions people could solve their needs using hardwood from gates to even instruments. Nowadays we have to rethink our attitude because of the lack of sources and global effects on Earth.

While we could use wood as a material to satisfy our needs – we lost control with plastic materials. It is time to look back and realize: we have to change our perspective. In earlier times people had the balance to live with nature: nowadays the balance needed to be restored. Thus, we have to adapt and improve by reusing wood and to use waste. Showing some examples is only a beginning – but to continue this way of thinking is the mission!

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## Properties of joists build by branches and twigs

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**Keywords:** architecture, joists, organic, ecology, folkish, branches, twigs

### ABSTRACT

Building with natural materials were the really first steps to mankind. The most optional and easy way was to use those resources which could be found on site. When the need appeared to have something else: forwarding came alive. Today's tendencies are to produce new materials and forwarding strengthened due to global economic reasons. The balance moved: natural resources cannot satisfy the needs of humanity – optimization and maximalization trying to solve this crisis (Kalpathy 2018).

Using those parts of woods which were not so used before hopefully can moderate the problem of not having enough resources. Several attempts and ideas came alive from reusing treated woods (Robert et al. 2006) to investigate specific structures.

In this paper I would like to acquaint some ways of investigating joists structures made by branches and twigs. This kind of thinking can lead us to natural balance and probably an affordable solution to today's crisis in building industry in the lack of resources. The most ideal state would be that situation, when these kind of branch and twig made structures could be used in real building situations: for example, while designing a family house (Sibel et al. 2015).

### INTRODUCTION

The ideology and attitude of today's architecture wants architects to rethink building materials which were 'forgotten', use more 'green' resources and reuse the existing ones to minimize the energy needs while producing them. Nowadays (global crises and sudden population growth) we have similar problems as we had earlier (world wars, natural disasters): the lack of (natural) sources.

Centuries ago, it was obvious to build a simple house from trees and stones – although nowadays we cannot afford this: our houses are built from prefabricated, source (material) minimalized solutions.

We have to rethink our goals and possibilities. I think today's architects' biggest mission is to help nature (full habitation) and to equalize the differences between nature and mankind.

Based on the facts mentioned above we will concern on some examples brought from the past and on those ones we have today to note: building with branches and twigs we can create structures for housing needs, too.

Based on earlier traditional (Hungarian) folkish solutions several options will be examined while looking for the ideal joist system for designing a family house made by branches and twigs. From the really basic system to the really complex one depending on the needs we want to solve with them.

This theoretical study is an entry to the real investigations: the goal of this study is to build several 1 m<sup>2</sup> floor area test joists and investigate them to several impacts (mechanical strength, fire resistance etc.).

After these experiments an overall guide would be made to architects in the first way: what kind of joists of branches and twigs can be designed and what is the limit of these specified structures?!

### SIMILAR ESSENTIAL (HUNGARIAN FOLKISH) STRUCTURES

Starting the investigation is obvious to study those systems which were made centuries ago and could be used nowadays, too. Because of this the examples beneath will show us how (mostly) peasants have been built their houses from those kinds of resources which were available in the surroundings.

### Wooden beam ceiling system

The most optional way of making a horizontal supporting system was making it using wooden beams (Fig.1). Beams were taken next to each other closely, due to this it was a resource intensive solution. Good properties of carrying loads and thermal insulation.

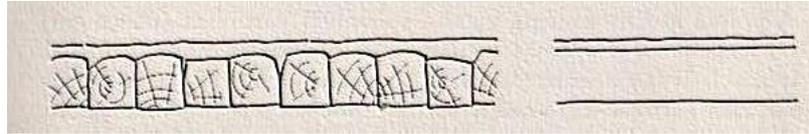


Figure 1: Wooden beam ceiling system (Mednyánszky 2020a)

### Wooden board ceiling system

Material saving solution. The main (holding) structure is the wooden beam placed 60-90 cm from each other by the middle point horizontally (Fig. 2). The boards were about 3,0 cm thick originally.

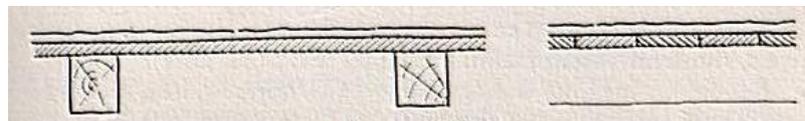


Figure 2: Wooden board ceiling system (Mednyánszky 2020b)

### Ceiling system made by laths rolled with swaddling clothes (clay+chaff)

Can not hold so much load than the other versions, making it takes more time but much more affordable than the previous variants (Fig. 3). Good thermal insulator, light weighted system. The top layer filled with sand and clay.

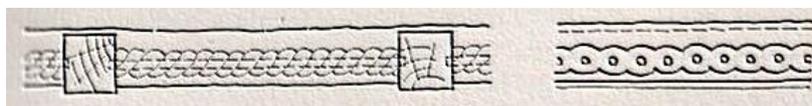


Figure 3: richly decorated passenger gate, note this different symbol (Mednyánszky 2020c)

### Ceiling system made by twig or reed

A solution which can be built fast and cheap (Fig. 4). Light weighted structure, the reed variant could be found near lakes and rivers due to clay resources. The top layer filled with clay in 10 cm of thickness.

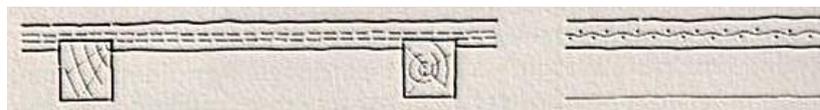


Figure 4: Ceiling system made by twig or reed (Mednyánszky 2020d)

## INVESTIGATIONS OF TODAY'S BRANCH AND TWIG SYSTEMS

The investigations are made on a 1m<sup>2</sup> floor area branch and twig systems in varied structural layers. The thickness and the orientation of the components (branches and twigs) are changing as well as the materials used as layers on the top of the ceiling structure (Fig. 5).

The acoustic-, thermal-, and burning properties investigated in the same time, too.

For an example the image shows several materials as layers used in the test objects.

R1:

- clay+sand+wooden boards
- ceiling system (branches and twigs are made in the same direction)

R2:

- light-weight concrete (polystirol)+reinforced plasterboard floor system
- ceiling system (branches and twigs are made in the same direction)

R3:

- wooden board floor system
- changed in orientation made branch and twig ceiling system

After the test results (maximum of mechanical pressure, torque etc.) the several systems can be known how far they can be loaded. Timing the fire resistance and the height of the noise there are also other properties we will know about these structural systems.

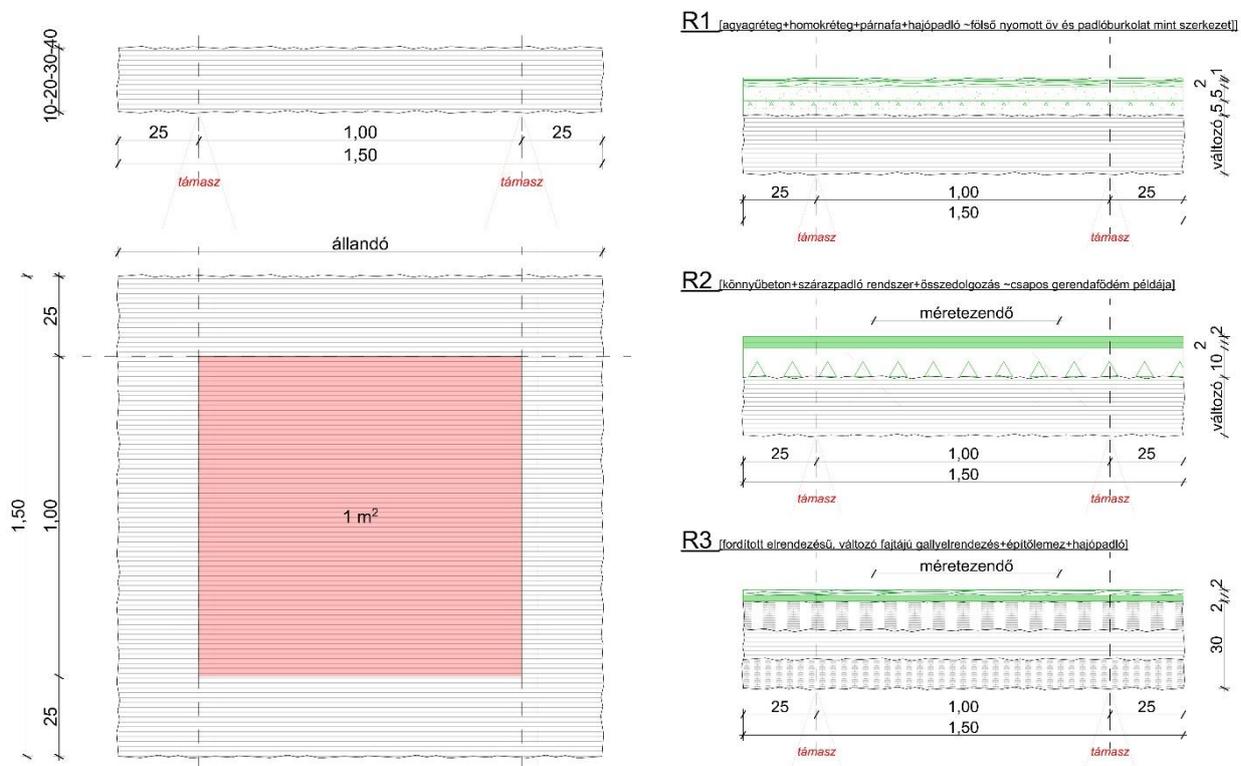


Figure 5: Thickness and orientation of components of the ceiling structure

## CONCLUSIONS

Based on early structures it can be noticed we can use waste materials: branches and twigs, too. Studying these structural systems, we can optimize them to satisfy today's housing needs. There is no need to (re)create materials and because of this the World's global economy could be restored by slowing non-reusable material usage.

Depending on several properties (thickness, diameter, wood type etc.) after testing these solutions and optimization, a guide can be made to architects and engineers, too.

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## Dynamic mechanical response of beech (*Fagus sylvatica* L.)

### Numerical analysis

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**Keywords:** tree biomechanics, finite element method, modal analysis, dynamic response

### ABSTRACT

The wind gusts occur more frequently in recent years and also hardwood stands can be seriously damaged by the wind storms. The mechanical responses of a tree at the forest scale are described by simplified models, for example FOREOLE (Ancelin et al. 2004), GALES and HWIND (Gardiner et al. 2000). These models describe static beam response with the use of a gust factor as a consideration of dynamic-wind characteristic. With the development of numerical methods, the more complex models of response to the turbulent airflow reflecting vibrations and damping of trees are available mainly for softwoods (Sellier et al. 2008). The experimental analyses of tree dynamic response are then more aimed at the effect of tree architecture, rather than considering tree defects (James et al., 2006).

Our work aims to present a modal, harmonic and transient finite-element analysis of broadleaved tree response with respect to its material properties and architecture. The analyses were carried out using ANSYS Mechanical APDL software to describe the natural frequencies, mode shapes and development of amplitudes in frequency and time domain. The parametric beam based finite-element model of the tree with orthotropic material was developed. The model outputs were compared with the field experiment data. The parametric definition of the model enabled “what-if” analyses with the change of geometry and material constants simulating different defects and trees scenarios. The relationship between the dynamic response of model and tree defects were found to be a significant, which provides the possibility of application in non-destructive tree assessment.

### INTRODUCTION

The increasing effect of forest disturbances is observed in the last decade of the century (Seidl et al., 2014). As part of these disturbances, over 50% of primary damage to the European forest was caused by wind storms (Gardiner et al., 2013). The wide range of studies and models has been developed over last twenty years to describe the mechanical response of trees to wind as main tribute of its dynamic loading (James et al., 2014). The majority of models which works on the forest scale are based on combination of mechanistic and statistical approach of static response and description of gust wind (Gardiner et al., 2008; Kamimura et al., 2019). However, these models mainly aimed on coniferous trees works on the principle of static behaviour and include analysis of the effect of storm and wind-flow (Kamimura et al., 2008).

Regardless to numerous studies focused on the description of wind characteristics in canopy models (de Langre, 2008), the detail description of individual tree response provide an important part in understanding of wind-tree interaction in stands. The dynamic response of structure can be observed in time or frequency domain. Time domain provide complex tree reaction (inclination, displacements, strains) (James et al., 2014) over time, while frequency domain provides general information of tree vibrational properties (Niklas, 1992). If the gust wind has similar frequency as the eigenfrequency of tree, the resonance occurs and can be followed by tree failure (Niklas, 2010). The eigenfrequency of trees has been intensively observed since 70's (Mayhead, 1973). Moore and Maguire (2004) provided extensive overview on coniferous and proved a strong correlation of frequency to ratio of diameter at breast height and squared tree height (DBH/H<sup>2</sup>). Bunce et al. (2019) confirmed this correlation for stands in temperate deciduous

forest. While in the case of large open grown deciduous trees the significant correlation was found with cumulative diameter of primary branches (Kane et al., 2014). The individual morphology influenced by surrounding conditions (open-grown or forest trees) is then a significant part of tree response in the frequency domain.

Because of the high correlation, the recent studies of tree oscillations are more concerned about the morphology. However the material properties, commonly describe by elastic parameters, influence the tree response also (Bunce et al., 2019; Sellier and Fourcaud, 2009). The significant change of material properties within a tree is usually caused by decay and it is related to the increase of risk of failure. The numerical study of Maritime pine (Yang et al., 2020), describe tree response mainly in time domain with simulation of failure. Model showed that in the case of change of root-soil conditions (anchorage stiffness) there is difference in oscillations between stem and crown. With change of root properties there was observed a slight decrease in 1<sup>st</sup> frequency during simulations. The change of properties in stem was followed by the failure in the case of gust wind simulation. The study also suggests that the position for observation of dynamic response can be important. The observation of higher frequencies could provide a better understanding of tree response too. Various authors agree that signal in frequency domain is noisy indicating number of higher frequencies caused by oscillation of branches (James et al., 2006), and that the second mode of vibration is usually very close to the first one. Kovacic et al. (2018) described two natural frequencies and damping ratios in two perpendicular trajectories of oscillating stem.

The aim of the following work is to provide detail analysis of tree dynamic response in time and frequency domain in relation to stiffness change representing defects of stem and root-plate system. The presented paper is a proof-of-concept study covering validation of experimental approach, building the finite element model (FEM) and illustration of relation between frequency domain and tree stiffness.

## METHODS

### *Measurement*

The pull and release test was performed on young beech (*Fagus sylvatica* L.) to validate the results of numerical analysis. The tree grows in small open area on the edge of the forest on nutrient-rich site with clay soil. The tree height is 6.1 m and stem diameter at the base is 9.5 cm. Stem and branches were divided into the segments to describe the tree structure. Diameter in two perpendicular directions of bottom and top part of each segment was measured together with length of segment, angle and azimuth of branching as well. The upper part of tree, which was not possible to access on the site, was analysed from two perpendicular photos. Measured structure of the tree was used as input for a finite element model (FEM). To induce a dynamic response the tree was pulled and released in short time period (up to 3 s) at the height of 1.8 m for 10 times. Three device types were used for the capturing of response: small accelerometers, big accelerometers and vibrometer. Three small 1-axis (MMF KS94B.10, sampling frequency - sf 20 kHz) and three big 3-axis (MMF KS813B, sf 10 kHz) piezoelectric accelerometers were placed along the stem. The accelerometers were placed in pairs on three positions: upper 3.73 m (AC 01), middle 3m (AC 02) and lower 0.95 m (AC 03) (Fig. 1a). The AC 01 was placed to the highest accessible position, AC 02 were placed to the position marked as anti-node in FEM modal analysis. AC 03 was placed in the middle part of stem without branches to observe the overall tree reaction. The laser Doppler's vibrometer (PDV-100, sf 20 kHz) was placed in the opposite of pulling direction. The vibrations were scanned in 2 m height above ground level. Camera (Canon EOS 700D, 18M 5184x3456) was placed perpendicularly to the pulling direction to capture a maximal displacement of stem (image analysed by correlation in Mercury RT2.7 sw.). Data from big accelerometers were collected by data-logger DEWE 43 (Dewesoft 7.0) and small accelerometers by DAQ DEWE 41 (Dewesoft 6.6.2). The measured data were analysed in time and frequency domain (by discrete Fast Fourier Transformation FFT) in MATLAB (R2020a).

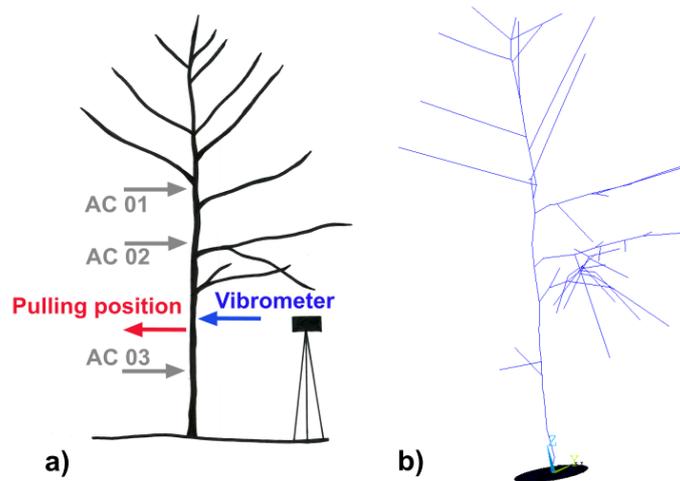


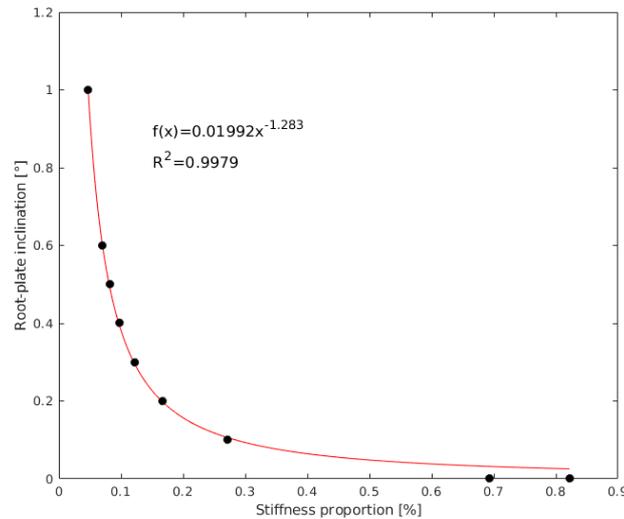
Figure 1: a) Scheme of measurement by AC01-03 accelerometers located at 0.95 m, 3 m and 3.73 m level; b) beam geometry of FEM.

### Numerical model

The finite element model (FEM) of tree dynamic response was build and analysed in ANSYS MECHANICAL APDL (19.2). The **geometry** of presented model was set according to the measurement. The original tree shape was simplified into the beam and shell structure. The simplification was chosen as a compromise of precision and demands on computation time and memory. Tapered beams (element type BEAM 189) were used for stem and branches. Root-plate was represented by circular area discretized by element type SHELL 281 (Fig. 1b). Stem and branches were created according to the measured segments. Elliptical cross-section has been assigned to top and bottom part of each segment. The crown of FEM was built up to the 2<sup>nd</sup> order of branches. Root-plate was created as circle divided into three rings (concentric layers), which allowed to define different ratio of soil and roots volume based on the distance from the stem. The diameter of the first layer was chosen to be two times higher than diameter of the stem base, second layer four times and third layer ten times higher, whereas last layer represented pure soil. The whole FEM is anchored in all tree directions on outer edge of the last layer.

Isotropic **material model** was defined by three elastic characteristics (longitudinal elastic modulus  $E_L$ , Poisson ratio  $\nu$  and density  $\rho$ ). The material properties for stem and branches (11.6 GPa for  $E_L$  and 965 kg/m<sup>3</sup> for  $\rho$ ) were set up according measurement of green wood samples (86 pcs.; 20x20x300 mm) from five beeches by frequency resonance method. The value of poisson ratio ( $\nu$ ) was defined as 0.45 according Hearmon (1948). The density of the 2<sup>nd</sup> order branches was increased by 15% to cover the added mass of leaves.

Material properties of root-plate were defined as a composite of roots and soil by rule of mixture. The proportion of roots was reduced with the distance from stem and last layer was represented only by soil properties. The elastic moduli for soil was set up on 20 MPa, density on 2000 kg/m<sup>3</sup> and Poisson ratio on 0.3 according Dupuy et al. (2007). Subsequently, the overall stiffness of the root-plate system was mostly tuned by the modification of the root properties. The representation of root-plate by the simplified model with shell elements required reduction of root-system stiffness to correspond with a real range of inclination values (Fig 2). The global model of root-plate was consequently used to describe the change of eigenfrequency (natural frequency) with a root-system stiffness changes. Therefore the three levels of stiffness (original stiffness, moderately reduced stiffness and significantly reduced stiffness) were analysed by modal analysis in FEM. The effect of stem stiffness to eigenfrequency was analysed also on three levels (original stiffness, moderately reduced stiffness and significantly reduced stiffness) by modal and harmonic analysis. In addition, the effect of branches on dynamic response was observed by reduction of FEM tree structure to one with 1<sup>st</sup> order branches and to clear stem without branches.



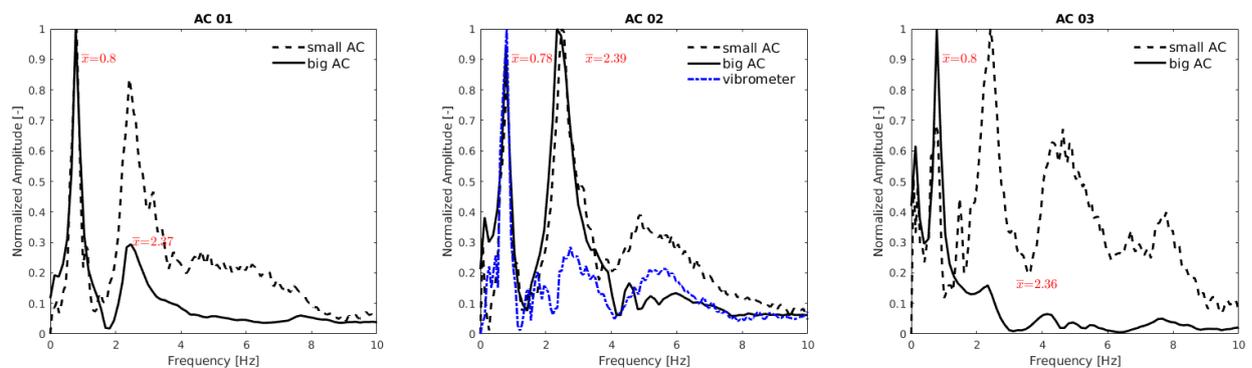
**Figure 2: Relation between root-system stiffness proportion and inclination for FEM shell structure of root-plate used for adjustment of stiffness analysis.**

Modal (MA) and harmonic (HA) analyses enabled to obtain results in frequency domain within the reduced computational time. MA was computed for the first 30 frequencies, HA was computed for the range of 0 - 10 Hz with 3000 sub-steps. Transient analysis (TA) provided results in both time and frequency domain and was used for FEM validation too. In the TA the tree was loaded by maximal displacement (19 cm) for 0.01 s and released. Total time for computation was 7 s with division to 0.0005 s for each computational sub-step. The effect of gravity (9.81 m/s, MA, HA, TA) and damping (damping ratio 0.6, HA, TA) was also included in the analyses.

## RESULTS AND DISCUSSION

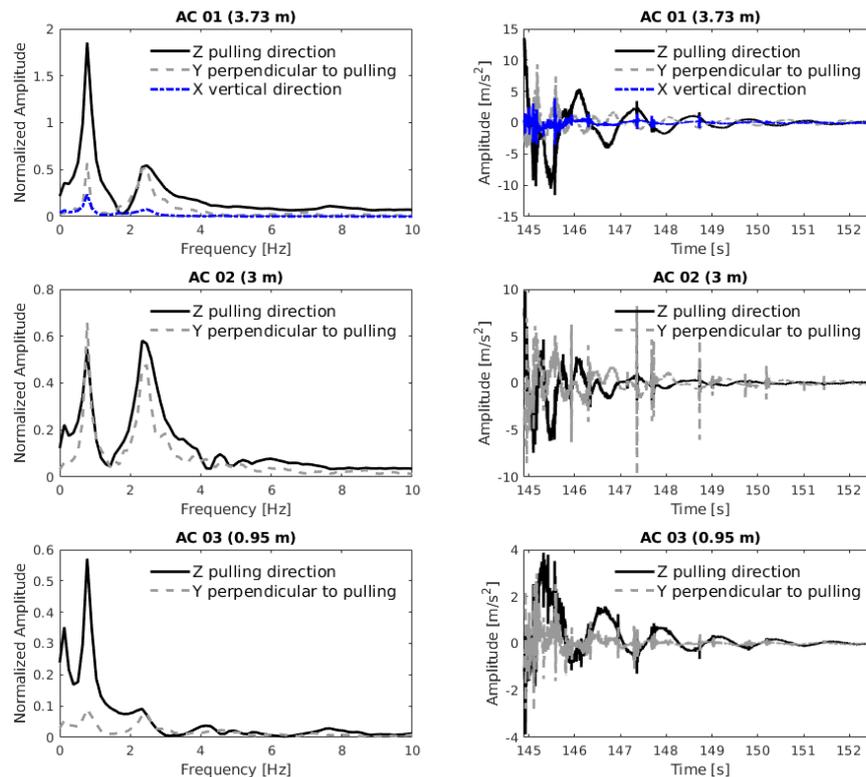
### Measurement and comparison of devices

All devices reliably measured 1<sup>st</sup> frequency (Fig. 3). Vibrometer provided slightly higher values (mean 0.81 Hz, var. coeff. 2.5%) in comparison to big accelerometers (mean 0.793 Hz, var. coeff. 5%) and small accelerometers (mean 0.797 Hz, var. coeff. 4.7%). Accelerometers provided also recognizable 2<sup>nd</sup> natural frequency (mean 2.4 Hz, var. coeff. 2%), although is more distinctive on the results from small accelerometers. There is also visible increase significance of higher frequencies with lower position along the tree height, what can be caused by the influence of branches above and confirms importance of measurement position (Yang et al., 2020).



**Figure 3: Measured frequencies from different device types in three positions: AC01 – accelerometers in 3.73m, AC02 accelerometers in 3m, AC03 accelerometer in 0.95m, vibrometer in 2 m (presented mean values are for the big accelerometers).**

Although the small (1-axis) accelerometers seem to be more sensitive to higher frequencies, the measurement was probably influenced by oscillation in perpendicular direction. The increase of higher frequencies is slightly evident in perpendicular direction for the big (3-axis) accelerometers (Fig. 4). The oscillation in vertical direction (Fig. 4 AC 01) is very low in comparison to other directions, therefore can be omitted during the measurement. In contrary, the significance of perpendicular direction increased for higher frequencies, especially for AC 02. The progress of amplitudes in time (Fig. 4 AC 01) shows phase shift between measured directions. This may also indicate presence of two frequencies for two oscillation axes closely to each other, what agrees with finding presented by (Kovacic et al., 2018a) and results from MA.



**Figure 4:** Comparisons of positions and directions of measurement in frequency (left) and time domain (right).

### **FEM validation and comparison of analyses**

The results of FEM dynamic response provided by TA was compared with measurement (Fig. 5) in time and frequency domain. 1<sup>st</sup> frequency of FEM were 7.6% lower and 2<sup>nd</sup> frequency 12.6% lower in the case of comparison of mean value from measurement and values from highest point in FEM (top of tree). The higher relative error (RE) than measured variation corresponds to the model simplification (for example rigid branch attachment, uniform  $E_L$ ) as described by (Sellier et al., 2006). These parameters were intentionally locked for the first set of analyses to eliminate the variability of inputs and needs to be investigated more in following work. The FEM is also more sensitive to higher frequencies, what can be observed in both (time and frequency domain, Fig. 5). The higher frequencies of FEM prevail in perpendicular and vertical direction. This can contribute to the knowledge of dynamic response since the measurement technique can damp higher frequencies in contrary to the outputs of numerical simulation. There was slight difference in 1<sup>st</sup> frequency for transient, harmonic and modal analyses (Fig. 6). The RE between TA and MA is 4.8% and between TA and HA 4.7%, although there were two close values between 1<sup>st</sup> (0.745 Hz) and 2<sup>nd</sup> (0.767 Hz) mode of MA. MA provided high number of frequencies. To identify a relevant frequencies (with higher amplitudes) for following analysis the HA was performed. From Fig. 6 is visible that HA corresponds to results from the TA in the whole spectrum despite provided a negligible higher 1<sup>st</sup> frequency and has more fine division in perpendicular direction.

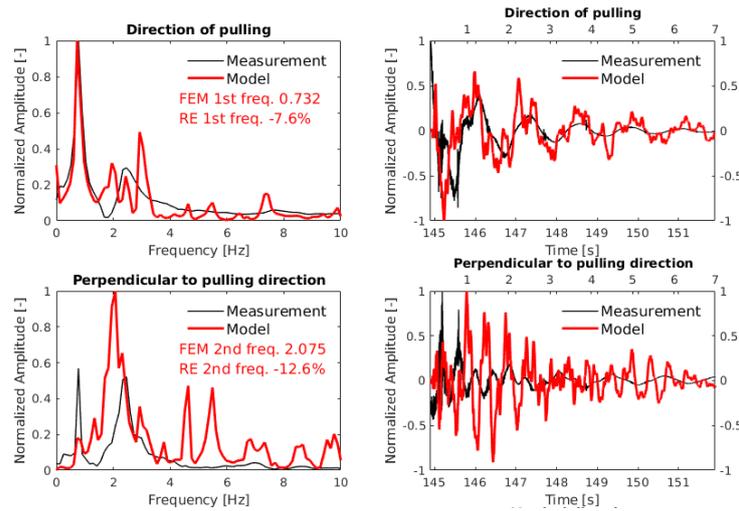


Figure 5: Comparison of FEM and measurement in frequency (left) and time (right) domain.

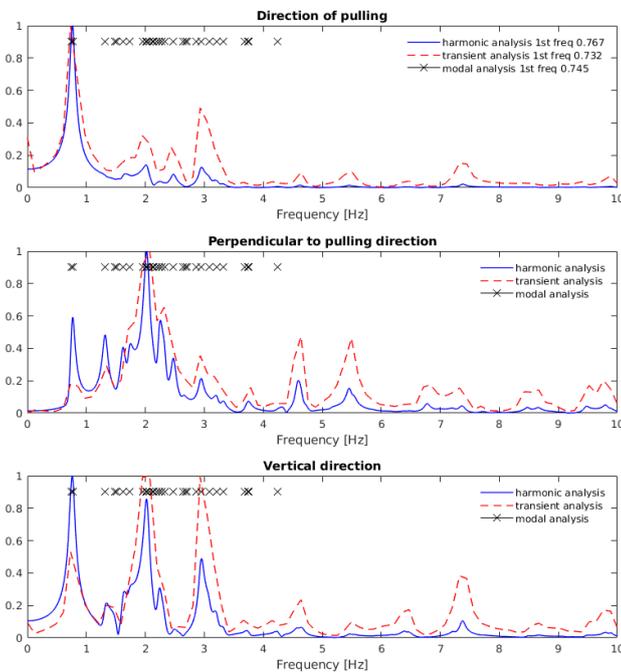
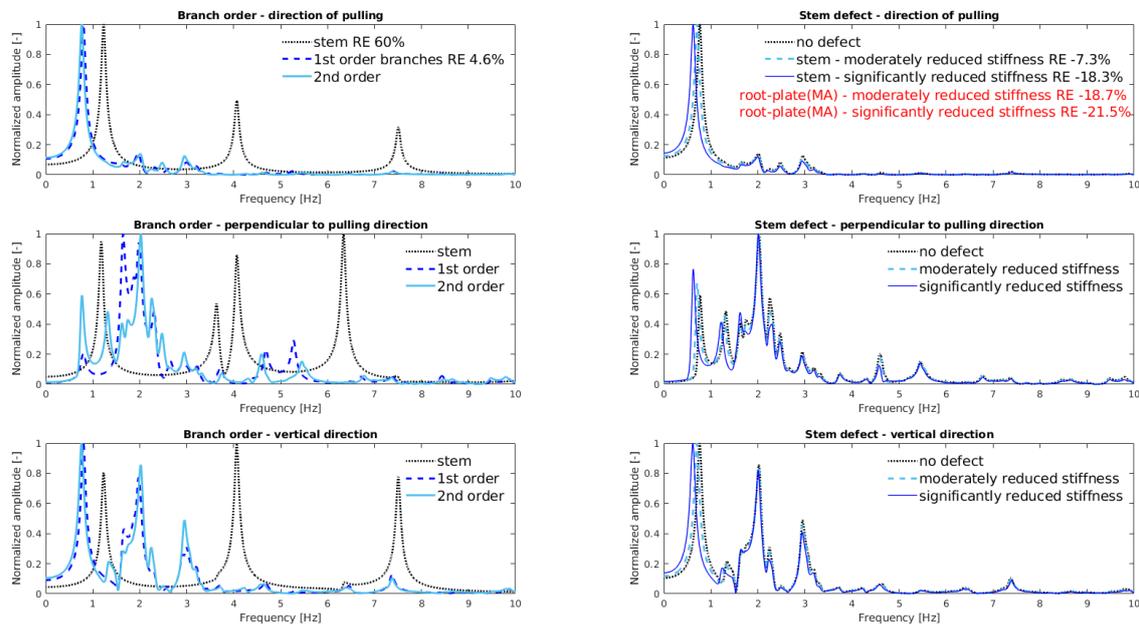


Figure 6: Comparison of modal, harmonic and transient analysis in frequency domain signal.

### What-if analyses

There is significant increase in 1<sup>st</sup> frequency (60%) for the clear stem (Fig. 7, left) compare to tree including branches. Next to change of 1<sup>st</sup> frequency there is also possible to see clear occurrence of another two higher significant frequencies (aliquots). With addition of 1<sup>st</sup> and 2<sup>nd</sup> order of branches there is shift of overall frequency spectrum to lower values and the decrease of higher frequencies amplitudes. This is probably caused by the additional branch damping and higher noise level for more complicated vibrating structure. The difference between frequencies for tree with 1<sup>st</sup> and 2<sup>nd</sup> order branches is lower, what is caused by the small difference of models (low amount of added branches for 2<sup>nd</sup> order). However the significance of overall tree structure to dynamic response is confirmed and corresponds to results of recent studies (Kane et al., 2014). The moderately reduced root-plate stiffness (analysed by the MA) caused 18.7% decrease of 1<sup>st</sup> frequency while significantly reduced stiffness caused 21.5% decrease, what implies a non-linear response. While the response in the case of stem stiffness reduction (7.3% for moderately reduced stiffness, 18.3% significantly reduced stiffness) is closer to the linear behaviour. The overall spectrum in the case of

stem defects is slightly shifted to the lower values (Fig. 7, right). In contrary to the different frequency spectrum in the case of different crown structure (Fig 7., left).



**Figure 7: Analyses of tree structure (left) and stiffness (right) influence the change in frequency domain obtained from harmonic analyses. RE is relative error of 1st frequency between original FEM and its variations. RE for root-plate stiffness are results of modal analysis.**

## CONCLUSION

Measurement of tree dynamic response provided two main natural frequencies from all used device types. The significance of measurement position and 2-axis measurement was shown for the observation of higher frequencies (more visible in lower part of tree and in perpendicular direction). The created FEM corresponds to measurement, but needs to be improved on the level of branch anchorage and material properties.

The harmonic, modal and transient analysis provided comparable result in frequency spectra. This confirmed reliability of less demanding analyses in the range of linear behaviour. Though the high number of out-of-scope frequencies from modal analysis showed on necessity of harmonic analysis.

The change of crown structure cause change in the frequency spectrum, while the stiffness reduction cause overall shift of frequencies. This could provide an option for the effect of defects observations. The higher frequencies in both horizontal axes could help to differentiate between effect of crown structure and tree stiffness changes.

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## The effect of different moisture contents on different mechanical properties of selected wood species

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**Keywords:** Moisture content, bending strength, compressive strength, Brinell-Mörath hardness

### ABSTRACT

Several influencing factors affect wood properties. The structure of annual rings, inhomogeneity, porosity, moisture content (*MC*) and many other factors have great significance on properties. In this study, the *MC* of wood was tested, which highly affects most physical and mechanical properties of wood (density, strength, elasticity, pliability, decay-resistance, electrical and thermal properties, anisotropic properties, shrinkage and swelling, etc.). Water can be found in wood in three states:

- liquid water and water vapor in cell lumens (free water)
- water bound in cell walls (bound water)
- crystallized water linked to the chemical components of wood.

Fiber-saturation point (*FSP*) is a specific *MC*, when all the intermicellar and interfibrillar cavities of the cell walls are saturated with water, but no free water can be found in the lumina. *FSP* depends on the wood species, usually 30% *MC* is accepted. Between the absolutely dry state and the *FSP*, both air and water can be found in the wood tissue and the mechanical properties of wood highly change with the *MC*. Over the *FSP* the mechanical properties of wood remain the same. The aim of this study is to determine the changes of bending strength, compressive strength and Brinell-Mörath hardness between the *MC*s of 12% and *FSP* for selected wood species: robinia (*Robinia pseudocacia*), sessile oak (*Quercus petraea*), beech (*Fagus sylvatica*), poplar (*Populus*), larch (*Larix decidua*) and spruce (*Picea abies*), and to compare them.

### INTRODUCTION

The properties of wood are the result of several influencing factors. The structure of annual rings and the resulting inhomogeneity, the porosity, the moisture content (*MC*) and many other factors have great significance on its properties, and these can be never ignored. In this study, the *MC* of wood was selected as main factor, which highly affects most properties of wood - density, strength, elasticity, surface treatment, pliability, decay-resistance, electrical and thermal properties, anisotropic properties, shrinkage and swelling, etc. (Molnár 2004).

Water can be found in wood in three states: liquid water (free water) and water vapor in cell lumens, water bound in cell walls (bound water), and crystallized water linked to the chemical components of wood. The fiber-saturation point (*FSP*) is the *MC*, when all the intermicellar and interfibrillar cavities of the cell wall are saturated with water, but no free water is found in the cell lumen *FSP* is at about 30% *MC*, depends on the wood species. Between the absolutely dry state and the *FSP*, both air and water can be found in the wood tissue. The mechanical properties of wood highly change, but over the *FSP* the mechanical properties of wood remain the same. Over *FSP*, free water is already present in the cell lumens and if the wood reaches its saturated state, no air remains in the lumens. (Skar, 1988)

The aim of this study is to determine the differences of bending strength, compressive strength and Brinell hardness between 12% *MC* and *FSP*.

## EXPERIMENTAL MATERIALS AND METHODS

During the described researches the mostly used Hungarian hardwood and softwood species had been selected: robinia (*Robinia pseudocacia*), sessile oak (*Quercus petraea*), beech (*Fagus sylvatica*), poplar (*Populus*), larch (*Larix decidua*) and spruce (*Picea abies*). The porosity of wood correlates well with its density ( $\rho$ ) at 12% MC (Tiemann 1906, Molnár 2004, Molnár et. al. 2016):

- high density ( $>700 \text{ kg/m}^3$ ): robinia, beech
- medium density (550-700  $\text{kg/m}^3$ ): oak, larch
- low density ( $<550 \text{ kg/m}^3$ ): poplar, spruce

Another important factor was that the selected species included both diffuse-porous (beech, poplar) and ring-porous wood species (oak, robinia). Only faultless samples with narrow annual rings had been tested, thus with homogeneous structure.

Prior to the measurements, the samples had been prepared according to the standards, and the tests were conducted according to the standards as well. The samples were divided into two equal groups. The first group was put in a climate chamber at a temperature of 20 °C and a relative humidity of 65%, to have a MC of 12%. The other group was soaked into distilled water to have a MC over their FSP.

### **Bending strength**

Both in the furniture industry and at the wood constructions, bending strength is an outstanding type of strengths, let us just think on roof-beams or on Thonet chair No. 14. In case of a three-point bending test for wood, the value of bending strength ( $\sigma_b$ ) is calculated using the Navier-equation corrected by Tanaka as follows (Eq. 1):

$$\sigma_b = \frac{F_{max}}{A} \quad (1)$$

where  $F_{max}$  [N] is the maximum force during the test and  $A$  is the cross-section of the sample in  $\text{mm}^2$ . The samples were produced as described in the standard ISO13061-03, with a size of  $20 \times 20 \times 300 \text{ mm}$  (Molnár 2004).

### **Compressive strength**

The resistance of wood to the load parallel or perpendicular to the grains is called compressive strength. Since compressive stresses often appear in wood structures as well as in many wood products, the significance of this mechanical property is not negligible. Compressive strength ( $\sigma_c$ ) is calculated using the following equation (Eq. 2):

$$\sigma_c = \frac{F_{max}}{A} \quad (2)$$

where  $F_{max}$  [N] is the maximum force during the test and  $A$  is the indented area of the sample in  $\text{mm}^2$ . In our tests, compressive strength parallel to the grains was investigated as described in ISO13061-17 (Molnár 2004, Kovács 1979).

### **Brinell-Mörath hardness**

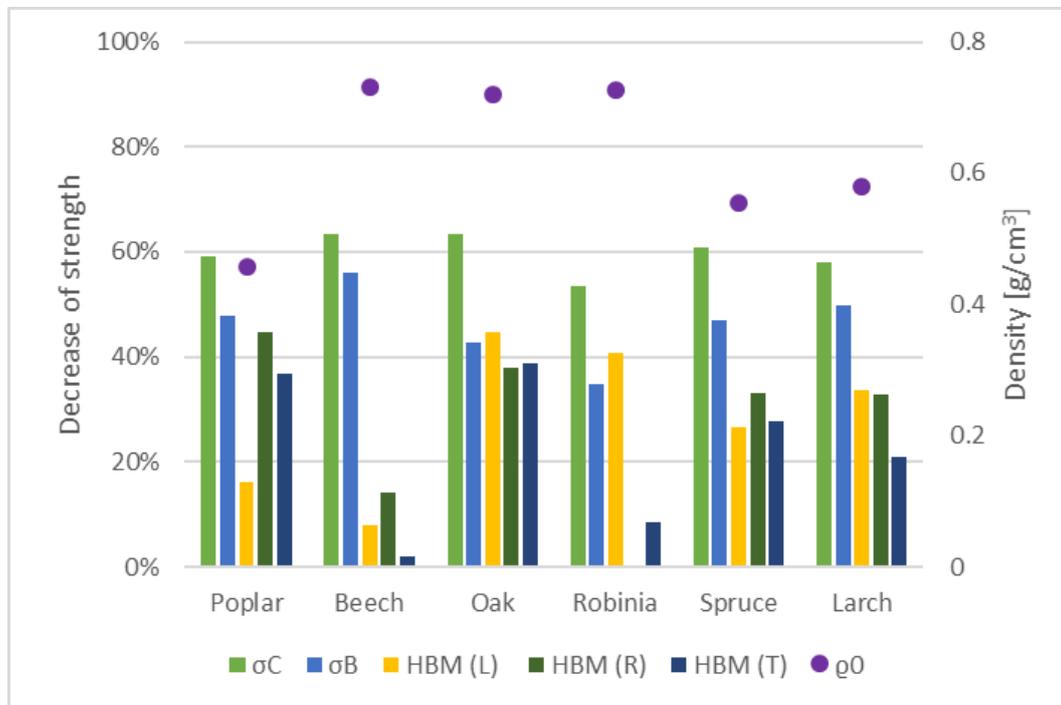
Hardness is the resistance of the material against the penetration of a tool. In 1900, Brinell expressed the hardness as the ratio of the load and the surface impressed by the 10 mm diameter ball. According to Mörath's amendment, very hard wood species are exposed to 1000 N, semi-hard wood species are exposed to 500 N and very softs are exposed to 100 N, as it is shown in the standard MSZ6786/11-82. The Brinell-Mörath hardness ( $H_{BM}$ ) is calculated by the next equation, using the depth of indentation ( $h$ ), the diameter of the ball ( $D$ ) and the diameter of the remaining impression ( $d$ ) (Eq. 3) (Vörös and Németh 2018a, 2018b):

$$H_{BM} = \frac{F_{max}}{D\pi h} = \frac{2F}{D\pi(D - \sqrt{D^2 - d^2})} \quad (3)$$

## RESULTS AND DISCUSSION

### **Strength decrease**

We considered that it is important to describe the extent of the decrease in strength caused by the decrease in moisture content globally before dealing with each type of wood or wood species separately. Strength decrease is shown in Figure 1.



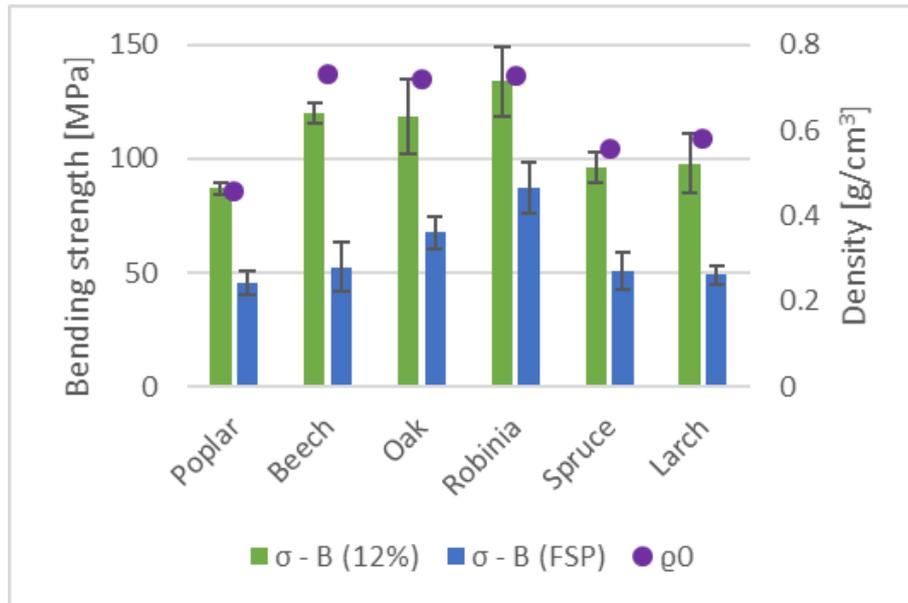
**Figure 1:** The oven-dry density and the decrease of both compressive strength and Brinell-Mörath hardness between 12% MC and FSP of selected wood species. Abbreviations:  $\sigma_c$  is the compression strength,  $\sigma_b$  is the bending strength,  $H_{BM}$  is the Brinell-Mörath hardness in longitudinal (L), radial (R) and tangential (T) direction and  $\rho_0$  is the dry density

The most drastic decrease can be observed in all cases at compressive strength (ca. 60%) and the smallest change at Brinell-Mörath hardnesses in the lateral anatomical directions (HBMR and HBMT) (ca. 2-40%), although the extent of the latter one highly differs.

It is clear that the degree of strength decrease does not depend on the density of wood. This observation is presumably mostly related to anatomical features. For example, changes in bending strength, compressive strength, and Brinell-Mörath hardness (L) varied nearly equally by the groups of wood species. The decrease of compressive strength, bending strength and Brinell-Mörath hardness in the longitudinal direction for poplar and beech was about 60%, 50%, 15%, for oak and robinia 55%, 40%, 40%, and for spruce and larch 60%, 50%, 30%, respectively.

### **Bending strength**

Changes in bending strength are shown in Figure 2.

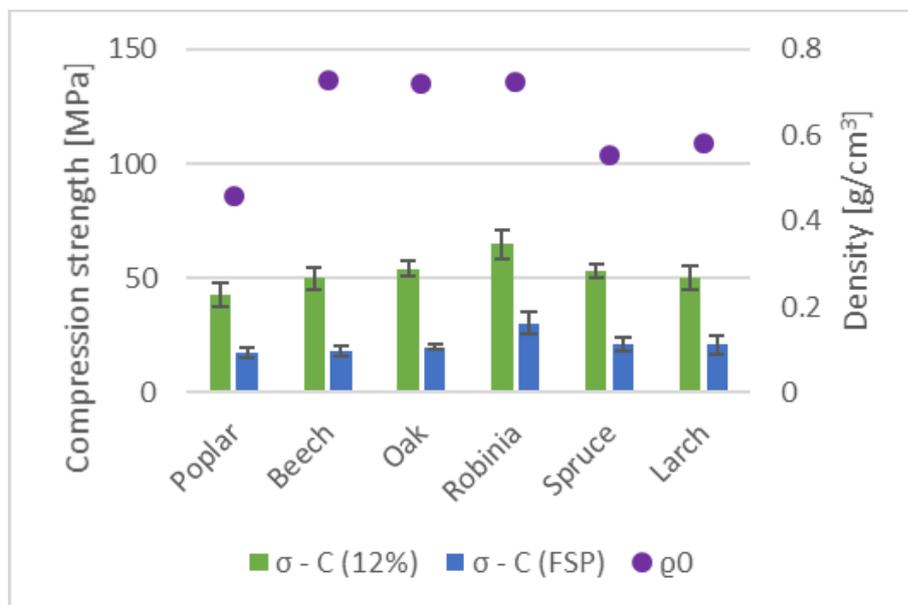


**Figure 2.** Changes in bending strength depending on the moisture content of spruce, larch, robinia, oak, beech and poplar. Abbreviations:  $\sigma - B$  is the bending strength at 12% MC (12%) and at fibre saturation point (FSP) and  $\rho_0$  is the dry density

As expected, the bending strength of samples with 12% MC correlate well with density. No such good correlation can be observed for samples with a MC over FSP. Despite the nearly same strength values and density of beech, oak and robinia, there is a significant difference in bending strength values over FSP. Except beech, all of the selected wood species showed a decrease of about 50 MPa as a result of the high increase in MC (Figure 2).

### Compressive strength parallel to the grain

The compressive strength values obtained for the examined wood species are described in Figure 3.



**Figure 3:** Changes in compressive strength depending on the moisture content of spruce, larch, robinia, oak, beech and poplar. Abbreviations:  $\sigma - C$  is the compressive strength at 12% MC (12%) and at fibre saturation point (FSP) and  $\rho_0$  is the dry density

The 12% compressive strength values show the same trend as density, but with a different rate of change. For FSP there is practically no correlation with density, but it can be stated that for all tree species-groups can be found a decrease of about 30 MPa.

### Brinell-Mörath hardness

Brinell-Mörath hardness was examined in three main anatomical directions: longitudinal, radial and tangential (Figure 4).

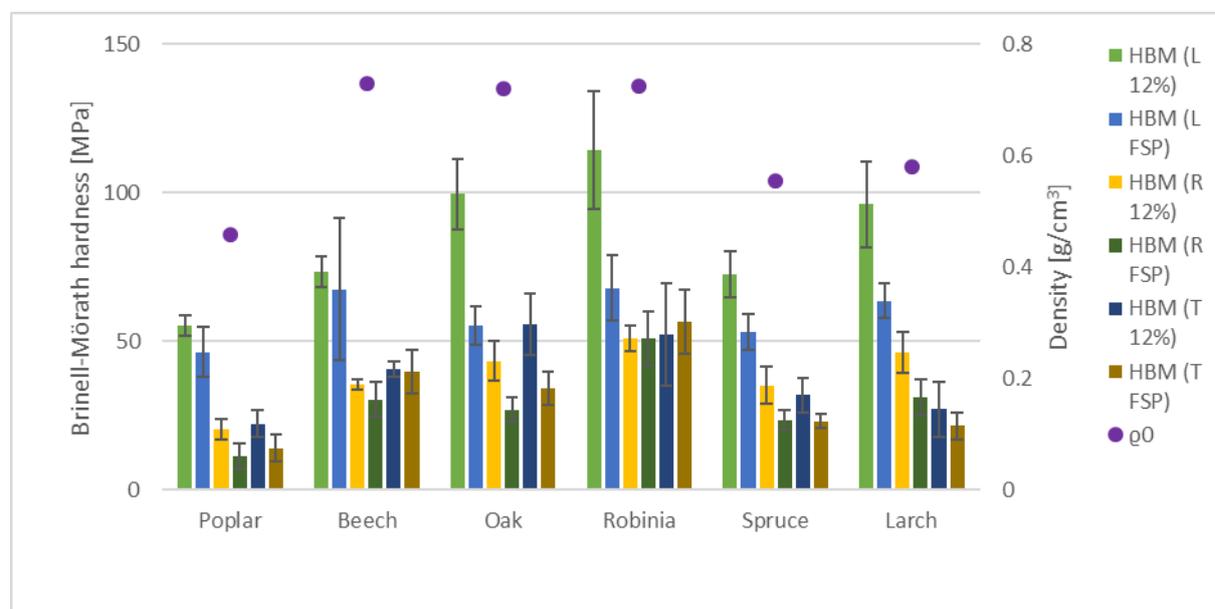


Figure 4: Changes in Brinell-Mörath hardness in 3 anatomical direction depending on the moisture content and density of spruce, larch, robinia, oak, beech and poplar. Abbreviations: *H<sub>BM</sub>* is the Brinell-Mörath hardness in longitudinal (L), radial (R) and tangential (T) direction at 12% MC (12%) and at fibre saturation point (FSP),  $\rho_0$  is the dry density

In the longitudinal Brinell-Mörath hardness of poplar and beech there is only a slight difference between the values of 12% MC and FSP. Both oak and robinia show a large decrease in strength, while for spruce and larch the decrease in HBML is about 30%. 12% R direction and 12% T direction took The values of HBMR and HBMT of poplar, beech, robinia and spruce are almost the same. within case of robinia, all lateral values are nearly the same. For larch, all examined values decreased about 25% between 12% MC and FSP.

## CONCLUSIONS

In our research we examined the compressive strength and bending strength of robinia (*Robinia pseudocacia*), sessile oak (*Quercus petraea*), beech (*Fagus sylvatica*), poplar (*Populus*), larch (*Larix decidua*) and spruce (*Picea abies*), moreover the Brinell-Mörath hardness in the three main anatomical directions. The tests were conducted on 12% moisture content and on fibre saturation point. All of these properties showed very different results for each test type and tree species, although the test results of samples with 12% moisture content typically correlated well with their oven-dry density. In order to achieve a more complex point of view regarding the examined properties, it is necessary to perform further studies.

## ACKNOWLEDGEMENTS

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