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# PREFACE

## New challenge of hardwood utilization

Hardwood is sometimes referred to the pearl of the forests. Hardwood utilization is more complex than softwood processing and there are great challenges for a competitive hardwood process chain. Due to many reasons such as changes in silviculture, changing ecological conditions the share of hardwoods in Europe's forests is growing and hardwood processing and utilization gains more and more importance. The overall objective of the proposed action is an improvement in competitiveness of European hardwood products by creating a Pan-European network with special emphasis on supporting early stage research.

A competitive and innovative hardwood process chain has to be developed by new allocation concepts to overcome the lack of economical efficiency of the mostly small enterprises with scattered hardwood resources, innovative manufacturing systems, a quality deployment model for specific process chains and concepts for technology transfer between research institutions and hardwood producers / processors has to be installed.

The conference series of hardwood research in Sopron, Hungary, already addressed the various assets and challenges of hardwood such as issues of hardwood research and utilization in Europe (first and second conference), the beauty of hardwood (third conference). Prof. Sándor Molnár and Dr. László Bejó did that time a pioneering work and established good reputation and scientific background for the conference edition in 2012.

We are delighted to present the proceedings of the 5th European Conference on Science and Technology on Hardwood, held in Sopron, Hungary, 2012, which has been organized in close cooperation with the University of Natural Resources and Applied Life Sciences, BOKU Vienna. This cooperation also reflects some cooperation in hardwood research of the two universities and we hope to present interesting topics of hardwood research and technology in the various sessions the industrial visit as well. We are grateful to all speakers, poster presenters and participants for this information transfer and for making this conference successful. We also hope that the readers of these proceedings will benefit from this documentation.

Alfred Teischinger & Róbert Németh

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# Relations between selected chemical properties and wood durability as well as mechanical features of thermally modified wood\*

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**Keywords:** thermally modified timber, chemical composition, mechanical properties, DPPH assay, wood durability

# ABSTRACT

Thermal modification of wood is an environmental friendly process for improving specific wood properties (color, durability, dimensional stability) without the use of additional chemicals. There is a constantly growing market for thermally modified timber, which challenges the industry to introduce faster and reliable methods for quality control and to eliminate some typical problems associated with the products and the technology (decreasing mechanical properties, emissions of volatile organic compounds from the wood, corrosion of technological equipment, waste water utilization).

The recent work focuses on three industrial technologies (with steam atmosphere, with inert gas atmosphere, and vacuum-press dewatering method) where optimization was carried out for the reduction of emissions by varying temperature and duration parameters. Beech, ash and spruce were considered for the research. Results are discussed regarding the changes in the loss of VOCs, pH of the wood extracts as well as the changes in the bending strength and the density of the wood.

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Regarding wood durability the mass loss was determined according to the EN 113 norm (incubation for 16 weeks). In connection with wood durability the total phenol content and the antioxidant activity of the wood were also measured using a novel, modified DPPH assay which was capable to measure the antioxidant ability of the wood directly instead of measuring the antioxidant level of the wood extracts only. Correlations have been established between durability parameters, total phenol content and antioxidant capacity. For beech and ash samples good correlations have been found between wood durability (mass loss) and DPPH assay values which could open up new possibilities for the estimation of wood durability in the future.

# INTRODUCTION

Thermal modification of wood is an environmental friendly process for improving specific wood properties (color, durability, dimensional stability) without the use of additional chemicals. It is usually carried out between 160 and 260 °C in various media (steam, nitrogen gas, vacuum, hot oil) using specific time schedules, depending on wood species and the properties which are to be attained (HILL 2006). There is a constantly growing market for thermally modified timber, which challenges the industry to introduce faster and reliable methods for the quality control of their products, especially in the field of determining wood durability, which has been also one of the main aims of the research recently (MILITZ AND ALTGEN 2011). Besides some decreasing mechanical properties e.g. bending strength and density (WINDEISEN ET AL. 2009) one major problem associated with thermally modified wood is its characteristic smell and the associated health issues. These emissions are measured as the loss of volatile organic compounds (VOCs), mostly acids, aldehydes and phenolic compounds (ROFFAEL ET AL. 2008, RÉTFALVI ET AL. 2009, WETZIG ET AL. 2011) which require process optimization to get rid of without compromising any of the desired features or worsening mechanical properties or increasing production costs significantly.

The recent work focuses on three industrial technologies (with steam atmosphere, with inert gas atmosphere, and vacuum-press dewatering method) where optimization was carried out for the reduction of emissions by varying maximum temperature and duration, varying the maximum temperature and pressure, and varying maximum temperature respectively. Beech, ash and spruce were considered for the research. Results are discussed regarding the changes in the loss of VOCs (acetic acid, formic acid, furfural, 5-methyl furfural), pH of the wood extracts as well as the changes in the bending strength and the density of the wood.

Regarding wood durability the mass loss was determined according to the EN 113 norm (incubation for 16 weeks). In connection with wood durability the total phenol content and the antioxidant activity of the wood were also measured. Antioxidant activity was determined using a novel, modified DPPH assay which was capable to measure the antioxidant ability of the wood directly instead of measuring the antioxidant level of the wood extracts only. Correlations have been established between durability parameters, total phenol content and antioxidant capacity. For beech and ash samples good correlations have been found between wood durability (mass loss) and DPPH assay values which could open up new possibilities for the estimation of wood durability in the future.

# **EXPERIMENTAL METHODS**

## Materials

Beech, ash and spruce were considered for research. All samples originated from industrial production. Table 1 summarizes the original parameters as well as the optimized parameters of the production for each technology. In the case of vacuum-press-dewatering technology only ash and beech was treated. As the samples originated from industrial production the exact parameters of the modification technology are not public.

Modification technology	Original parameters	Optimized parameters		
$N_2$ gas	Unknown	Pressure is released from the autoclave after reaching maximum temperature (purging).		
Steam	Unknown	Reducing the maximum temperature while increasing treatment time.		
Vacuum-press-dewatering	Unknown	Increasing maximum temperature from 195 to 210 °C with the same heating program and vacuum.		

Table 1: The modification technologies and the parameters

# Loss of VOCs and the pH of the wood extracts

The loss of VOCs was determined according to the flask method (DIN EN 717-3) after incubation for 24 hours at 40 °C. The extracts were filtered and their pH was measured using a potentiometric pH meter. The VOC components of the extracts were separated and quantitated using HPLC (Bio-Rad Aminex HPX-87H column, 50 °C, 0.005M  $H_2SO_4$  mobile phase at 0.6 ml/min and UV detection at 210 nm). Results were given in mg/g dry wood. One replicated was made from each sample.

# Total phenol content

100g of wood samples were ground and sieved. The 0.2-0.63 mm fraction was used for further analysis. 0.25 wood was extracted in 6 consecutive steps with 80% aqueous methanol in an ultrasonic bath. In one extraction step 4 ml of solvent was used for 30 minutes. Extracts were collected in a 25 ml volumetric flask and filled up after the final step. Total phenol content was determined using the method of Folin and Ciocaltau (SINGLETON AND ROSSI 1963) and was given in mmol quercetin/100g dry wood. The measurement was done in 3 replicates.

# Modified DPPH assay

DPPH assay was modified so as to measure the antioxidant capacity of the wood directly. 1 mg of wood (0.2-0.63 mm fraction) was measured in a plastic cuvette (1 cm optical path). After that a teflon-covered magnetic stirring bar (0.7 cm) was placed into the cuvette. 2.25 ml methanol was filled into the cuvette and then 0.75 ml 200  $\mu$ M DPPH solution was added. The cuvette was covered and placed on a magnetic stirrer (speed: 800 1/min) in a thermostated room, at a dimmed place. After incubation time (30 min) absorbance was determined at 515 nm. Using reference graphs the IC<sub>50</sub> value (50% inhibition value expressed in mg dry wood/ml assay) was determined. The measurement was done in 3 replicates.

# Bending strength and density

Bending strength was evaluated according to DIN 52186 norm on samples sized 400x20x20 mm conditioned at 20 °C and 65% relative humidity. Wood density was determined according to DIN 52182 on wood cubes sized 20x20x10 mm conditioned at 20 °C and 65% relative humidity using 20 replicates per sample.

# Wood durability

Mass loss of given sizes of wood specimens have been determined according to EN 113 norm after 16 weeks of controlled incubation with 6 replicates per sample. The used fungi were *Trametes versicolor*, *Coniophora puteana* and *Oligoporus placenta*. Mass loss is indicated in % and is related to dry wood mass.

# **RESULTS AND DISCUSSION**

## Main chemical features of the samples

Tables 2-4 summarize the main chemical parameters of the samples including untreated wood as a reference. Total phenol content reflects the transformation (cleavage) of lignin during the thermal treatment. The simple phenols which are produced during the cleavage of lignin contribute to the colour and to the durability (PFRIEM ET AL. 2009) and also to the characteristic smell of thermally modified timber. Most of these compounds are volatile and with increasing temperature and with the application of a vacuum they vanish from the wood: this can be tracked in the tables when comparing vacuum press dewatering technology to the other two processes. This is also true for the measured acids and furfurals.

Table 2: Chemical analyses for ash samples							
Technology	Total phenol [mmol/100g]	pH of extracts	Acetic acid loss [mg/100g dry wood]	Formic acid loss [mg/100g dry wood]	Furfural loss [mg/100g dry wood]	5-Methyl- furfural loss loss [mg/100g dry wood]	
Untreated	3.36	3.70	20	< 1	< 1	n/d	
N <sub>2</sub> gas original	13.58	2.80	1537	207	74.3	n/d	
N2 gas optimized	6.45	3.01	127	7	14.3	2.44	
Steam original	15.07	2.84	988	154	118.1	n/d	
Steam optimized	13.00	2.72	587	94	77.7	6.8	
Vacuum original	5.28	3.41	95	5	4.6	0.91	
Vacuum optimized	6.85	3.52	61	3	2.5	0.81	

Table 3: Chemical analyses for beech samples Total phenol pH of Acetic acid Formic acid 5-Methyl-Technology Furfural [mmol/100g] extracts loss loss furfural loss loss [mg/100g [mg/100g [mg/100g loss [mg/100g dry wood] dry wood] dry wood] dry wood] Untreated 1.85 3.72 22 < 0.5 < 0.6 n/d N<sub>2</sub> gas original 16.27 2.72 2378 212 176 n/d N2 gas optimized 2.92 3.06 170 18 7 2.73 112 215 Steam original 16.8 2.83 1156 n/d 14.02 Steam optimized 187 2.67 1066 86 6.21 Vacuum original 3.21 3.6 53 2 5 0.67 Vacuum optimized 5.11 3.68 48 3 3 n/d

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Table 4: Chemical analyses for spruce samples							
Technology	Total phenol [mmol/100g]	pH of extracts	Acetic acid loss [mg/100g dry wood]	Formic acid loss [mg/100g dry wood]	Furfural loss [mg/100g dry wood]	5-Methyl- furfural loss loss [mg/100g dry wood]	
Untreated	1.25	3.50	19	4.9	< 0.8	n/d	
N <sub>2</sub> gas original	3.14	2.94	430	100.0	66.4	n/d	
N2 gas optimized	2.32	3.02	99	48.0	32.4	0.73	
Steam original	5.40	2.96	511	119.0	81.9	n/d	
Steam optimized	2.15	2.84	332	125.7	108.6	2.46	

Considering optimized technological parameters it can be seen that applying a purging in case of  $N_2$  technology the loss of the volatiles decreases beneficially. The least effective optimization regarding reduction of the emissions is increasing duration and reducing temperature (steam technology) as the VOC losses decrease only slightly or even increase (Table 4, spruce samples). Best values are obtained altogether for the vacuum-press-dewatering technology as this is an opened system, by-products are sucked out from the autoclave continuously during the modification process: VOC loss values are close to the untreated (reference) values.

## Main physical features and selected chemical properties of the samples

Tables 5-7 summarize the main physical parameters and some selected chemical features of the samples including untreated wood as a reference. Both IC<sub>50</sub> value and total phenol content were included again, because their connection to wood durability is discussed further on. The purging of the volatile products with the N<sub>2</sub> technology proved to be successful for reducing VOC emissions however the wood durability parameters (mass losses) were deteriorated by this significantly. Products became less durable against *Trametes versicolor*, especially with beech. There is a great difference between the wood durability can be established with the vacuum-press-dewatering procedure, which must be due to the increased temperatures. There is also a diverse reaction for the wood density and bending strength regarding the optimization of the different technologies.

Technology	Mass loss Tram vers. [%]	Mass loss Con. put. [%]	Total phenol [mmol/100g dry wood]	IC <sub>50</sub> [mg dry wood/cm <sup>3</sup> ]	Density [g/cm <sup>3</sup> ]	Bending strength [N/mm <sup>2</sup> ]
Untreated	22.59	23.11	3.36	0.3563	0.664	124.31
N <sub>2</sub> gas original	2.86	0.20	13.58	0.2237	0.612	82.76
N2 gas optimized	13.10	1.43	6.45	0.3031	0.638	100.95
Steam original	11.31	1.98	15.07	0.2380	0.618	79.58
Steam optimized	10.82	0.85	13.00	0.2354	0.631	79.58
Vacuum original	19.07	5.95	5.28	0.2680	0.636	111.20
Vacuum optimized	6.41	2.04	6.85	0.3150	0.555	75.45

Table 5: Physical and selected chemical parameters for ash samples

Table 6: Physical and selected chemical parameters for beech samples						
Technology	Mass loss Tram vers. [%]	Mass loss <i>Con. put.</i> [%]	Total phenol [mmol/100g dry wood]	IC <sub>50</sub> [mg dry wood/cm <sup>3</sup> ]	Density [g/cm <sup>3</sup> ]	Bending strength [N/mm <sup>2</sup> ]
Untreated	21.02	33.09	1.85	0.6691	0.643	109.62
N2 gas original	5.90	0.50	16.27	0.2356	0.586	66.70
N2 gas optimized	27.94	22.48	2.92	0.5703	0.614	104.20
Steam original	10.86	2.04	16.80	0.2353	0.605	76.77
Steam optimized	22.47	3.73	14.02	0.6691	0.614	81.63
Vacuum original	19.07	5.95	3.21	0.5644	0.683	116.71
Vacuum optimized	6.41	2.04	5.11	0.2173	0.676	106.02

Table 7. Physical and selected chemical parameters for spruce samples

Technology	Mass loss	Mass loss	Total phenol	IC <sub>50</sub>	Density	Bending		
	Olig. plac.	Con. put.	[mmol/100g	[mg dry	[g/cm <sup>3</sup> ]	strength		
	[%]	[%]	dry wood]	wood/cm <sup>3</sup> ]		[N/mm <sup>2</sup> ]		
Untreated	25.22	24.51	1.25	21.81	0.464	87.71		
N <sub>2</sub> gas original	3.04	1.99	3.14	1.11	0.462	65.38		
N2 gas optimized	16.45	9.26	2.32	0.2363	0.480	78.59		
Steam original	7.64	4.45	5.40	0.4464	0.447	63.14		
Steam optimized	9.13	8.60	2.15	0.8373	0.462	59.03		

The  $IC_{50}$  values correspond to the antioxidant capacity of the wood samples. Recent studies have reported that the antioxidant capacity of thermally modified timber increases (AHAJJI ET AL. 2009) which could be attributed to the formation of stable free radicals. These radicals have proven to contribute to the durability of the thermally modified timber (WILLEMS ET AL. 2010). As phenolic compounds are powerful antioxidants their concentration can also be significant in this regard especially as these compounds are formed during the thermal modification process and their effect on the durability of thermally modified timber is well known (PFRIEM ET AL. 2009). Measuring the concentration of theses free radicals can open up new possibilities for the estimation of the durability of thermally modified timber with new methods in the future (MILITZ AND ALTGEN 2011).

## Correlations between wood durability and antioxidant ( $IC_{50}$ ) values

From Tables 5-7 it can be established that there is an apparent tendency between  $IC_{50}$  as well as total phenol content and mass loss (wood durability) values.

In this study total phenol content was determined from the wood extracts as simple phenolic compounds are well soluble in methanol and water. The whole antioxidant capacity was determined however from the wood itself, as non-extractable parts of the wood matrix (e.g. modified lignin structures) can also contribute to wood durability and these can not be extracted with neutral solvents. That is why the antioxidant (DPPH) assay was modified by us as

described in the "Experimental methods" section to be able to qualify the *antioxidant properties of the wood itself*.

Linear correlations have been set up between mass loss and  $IC_{50}$  as well as total phenol content values including "untreated", "original" and "optimized" values for each technology and for each wood species. Detailed results are included in Tables 8-10.

 Table 8: Correlations for ash samples between mass losses and chemical properties

Technology	Mass loss (Tram. vers.) versus IC <sub>50</sub>	Mass loss ( <i>Con. put.</i> ) versus IC <sub>50</sub>	Mass loss (Tram. vers.) versus Total phenol	Mass loss (Con. put.) versus Total phenol
N <sub>2</sub> gas	y = 147.2x - 30.51	y = 160.5x - 39.01	y = -1.844x + 27.23	y = -1.880x + 22.90
	$R^2 = 0.9916$	$R^2 = 0.6907$	$R^2 = 0.9596$	$R^2 = 0.5847$
Steam	y = 96.41x - 11.76	y = 181.51x - 41.56	y = -1.043x + 25.83	y = -1.961x + 29.19
	$R^2 = 0.9997$	$R^2 = 0.9993$	$R^2 = 0.9593$	<b>R</b> <sup>2</sup> =0.9559
Vacuum	y = 32.72x + 5.77 $R^2 = 0.0289$	y = 187.91x - 48.47 $R^2 = 0.5488$	y = -4.317x + 38.12 $R^2 = 0.8533$	$y = -5.932x + 40.72$ $R^2 = 0.9286$

Table 9: Correlations for beech samples between mass losses and chemical properties

Technology	Mass loss (Tram. vers.) versus IC <sub>50</sub>	Mass loss (Con. put.) versus IC <sub>50</sub>	Mass loss (Tram. vers.) versus Total phenol	Mass loss (Con. put.) versus Total phenol
N <sub>2</sub> gas	y = 42.77x - 2.74	y = 72.75x - 17.08	y = -1.30x + 27.42	y = -2.00x + 32.71
	$R^2 = 0.7434$	$R^2 = 0.9889$	$R^2 = 0.8633$	$R^2 = 0.9348$
Steam	y = 8.12x + 15.12	y = 66.41x - 11.50	y = -0.43x + 22.89	y = -2.17x + 36.67
	$R^2 = 0.1124$	$R^2 = 0.9868$	$R^2 = 0.3044$	$R^2 = 0.9839$
Vacuum	y = 3.21x + 15.71 $R^2 = 0.9259$	y = 21.61x - 1.25 $R^2 = 0.9987$	y = -1.02x + 21.48 <b>R<sup>2</sup>=0.7514</b>	$y = -7.52x + 39.45$ $R^2 = 0.9562$

 Table 10: Correlations for spruce samples between mass losses and chemical properties

Technology	Mass loss (Olig. plac.) versus	Mass loss (Con. put.) versus	Mass loss (Olig. plac.) versus Total phenol	Mass loss ( <i>Con. put.</i> ) versus Total phenol
	IC <sub>50</sub>	IC <sub>50</sub>		
N <sub>2</sub> gas	y = 0.710x + 9.41	y = 0.882x + 5.11	y = -11.56x + 40.75	y = -12.03x + 38.83
	$R^2 = 0.6051$	$R^2 = 0.8774$	$R^2 = 0.9619$	$R^2 = 0.9843$
Steam	y = 0.796x + 7.86	y = 0.852x + 5.96	y = -3.227x + 23.46	y = -3.88x + 23.91
	$R^2 = 0.9963$	$R^2 = 0.9675$	$R^2 = 0.5223$	$R^2 = 0.6411$

Generally there are better correlations between mass loss parameters and  $IC_{50}$  values than between mass loss values and total phenol content. Not only wood species but also the type of the technology is determinant in terms of the goodness of the correlations. Very tight fits have been established between  $IC_{50}$  value and mass loss for ash (*Trametes versicolor*) and for beech *Coniophora puteana* which could give basis for further method development.

# CONCULSION

Optimizing the technology for producing thermally modified timber is important regarding the reduction of emissions, obtaining the desired features (colour, durability, dimensional stability, etc.) while not compromising mechanical properties. The present work focused on three current technologies and three wood species investigating the effects of the temperature and pressure to obtain reduced emission values. Changing the parameters can result in the reduction of VOC emissions but care must be taken that this is not accompanied by the loss of mechanical parameters. A new method has been described to measure the antioxidant capacity of the wood directly and tight correlations have been found between wood durability (mass loss by fungi) and IC<sub>50</sub> values. The proposed method could be suitable for estimating wood durability parameters rapidly for a given wood species and for a given technology in the future. This requires however that the measurements be repeated on a larger number of samples.

# REFERENCES

AHAJJI, A., DIOUF, P. N., ALOUI, F., ELBAKALI, I., PERRIN, D., MERLIN, A., GEORGE, B. (2009) Influence of heat treatment on antioxidant properties and colour stability of beech and spruce wood and their extractives. *Wood Science and Technology*, **43**, 69–83.

HILL CAS (2006) Wood modification odchemical, thermal and other processes. Chichester, John Wiley & Sons.

MILITZ, H., ALTGEN, M. (2011) Development of methodology to predict wood properties of thermally modified wood. In: *COST Action FP0904* "*Thermo-Hydro-Mechanical Wood Behaviour and Processing*", February 16-18, 2011, Biel (Bienne), Switzerland. pp 17–18.

PFRIEM, A., HORBENS, M., BEYER, M., PETERS, M. (2009) Untersuchungen von Extraktstoffen aus thermisch modifizierter Rotbuche (*Fagus sylvatica* L.) auf ihre fungizide Wirkung. *Holztechnologie* **50**(2): 32–36.

RÉTFALVI, T., HOFMANN, T., NIEMZ, P. (2009) The environmental chemical features of waste water originating from the thermal treatment of wood. Wood Research, **54**(4):13–23.

ROFFAEL, E., KRAFT, R., NIEMZ, P. (2008) Formaldehyd- und Säureabgabe aus Thermoholz. *Holz-Zentralblatt* **134**(50): 1418.

WETZIG, M., HOFMANN, T., RÉTFALVI, T., SIEVERTS, T., BERGEMANN, H., NIEMZ, P. (2011) Physical, mechanical and chemical properties of wood, heat-treated with the vacuum-press-dewatering method. *COST Action FP0904 "Thermo-Hydro-Mechanical Wood Behaviour and Processing"*, February 16-18, 2011, Biel (Bienne), Switzerland. pp 35–36.

WILLEMS, W., TAUSCH, A., MILITZ, H. (2010) Direct estimation of the durability of high-pressure steam modified wood by ESR-spectroscopy, *IRG/WP 10-40508*, pp. 1–9.

WINDEISEN, E., BÄCHLE, H., ZIMMER, B., WEGENER, G. (2009) Relations between chemical changes and mechanical properties of thermally treated wood. *Holzforschung* **63**:773–778

# Selected physical and mechanical behaviour of sycamore maple (*Acer pseudoplatanus* L.) and European beech (*Fagus sylvatica* L.)

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**Keywords:** Mechanical properties, beech, sycamore maple, MOE, tensile strength, fracture toughness

## ABSTRACT

Physical and mechanical properties of European beech (*Fagus sylvatica* L.) and sycamore maple (*Acer pseudoplatanus* L.) were investigated as a basis for three-dimensional material modelling for structural simulations (e.g. with the finite element method). The mechanical properties of tensile (Young's modulus as well as Poisson's ratio, tensile strength) and fracture toughness were determined. The tests were carried out for different moisture content and also in all three main anatomical directions.

## **INTRODUCTION**

Beech (*Fagus sylvatica* L.) and sycamore maple (*Acer pseudoplatanus* L.) are common hardwoods in Central Europe (Brändli 2010).

Therefore, hardwood is used for conventional applications like parquetry, interior joinery, furniture and musical instruments (sycamore maple) and also more and more for wood constructions (glue lam). For glue lam, beech and ash are especially favoured. Thereby, well-founded knowledge of the material properties of these species is even more necessary (for example for calculation of boards with FE methods). Therefore, among the strength properties, the complete elastic parameter-set (3 moduli of elasticity (MOE), 3 shear moduli (G) and 6 Poisson's ratios) within the three main directions (longitudinal (L), radial (R) and tangential (T)) is required. Analogously, the influences of grain angle (LR, LT) and ring angle (RT) on the parameters mentioned above have to be known. Ideally, the parameters of the plastic deformation should also be known since this topic is increasingly studied (e.g. Schmidt 2008, Hering 2011).

Altogether, very complex measurements are required to allocate all essential data in a numerical simulation. The following parameters are necessary for a comprehensive material characterization:

- Elastic parameters (including properties on cyclic load and on hysteresis effects)
- > Strength
- Viscoelastic and plastic parameters
- Mechano-sorptive parameters
- Creep and relaxation
- > Thermal and moisture dependent behaviour

The total stress in an element under mechanical load and cyclic climatic conditions consists, according to Equation (1), of:

- Mechanical stresses
- Climatically induced stresses like shrinkage and swelling

The total strain is composed accumulatively as

$$\varepsilon_{\text{tot}} = \varepsilon_{\text{el}} + \varepsilon_{\text{m}} + \varepsilon_{\text{ve}} + \varepsilon_{\text{pl}} \tag{1}$$

where  $\varepsilon_{tot}$  is the total strain constituted of  $\varepsilon_{el}$  the elastic strain,  $\varepsilon_m$  the moisture induced strain,  $\varepsilon_{ms}$  the mechano-sorptive strain,  $\varepsilon_{ve}$  the viscoelastic strain and  $\varepsilon_{pl}$  the plastic strain.

Supplementary to this, the bonding, surface coating and overlay material as well as defects within the material, like cracks and burrowing passages of wood insects, influence the material behaviour.

The three-dimensional elastic behaviour of an orthotropic material can be described with the generalised Hooke's law. Equation (2) shows the compliance matrix using the engineering elastic parameters.

$\left[ \mathcal{E}_{11} \right]$		$\boxed{\frac{1}{E_{11}}}$	$\frac{\mu_{21}}{E_{22}}$	$-\frac{\mu_{31}}{E_{33}} \\ -\frac{\mu_{32}}{2}$	0	0	0	$\sigma_{11}$	
$arepsilon_{22}$		$\overline{E_{11}}$ $-\frac{\mu_{12}}{E_{11}}$	$\frac{1}{E_{22}}$ $\underline{\mu_{23}}$	$-\frac{\mu_{32}}{E_{33}}$	0	0	0	$\sigma_{22}$	
<i>E</i> <sub>33</sub>	_	$-rac{\mu_{13}}{E_{11}}$	$-rac{\mu_{23}}{E_{22}}$	$\frac{1}{E_{33}}$	0	0	0	$\sigma_{33}$	
Y <sub>23</sub>		0	0	0	$\frac{1}{G_{23}}$	0	0	$ au_{23}$	(2)
<i>γ</i> <sub>13</sub>		0	0	0	0	$\frac{1}{G_{13}}$	0	$\tau_{13}$	
$\gamma_{12}$		0	0	0	0	0	$\frac{1}{G_{12}}$	$\tau_{12}$	

where  $\varepsilon_{ii}$  are the normal strains,  $\gamma_{ij}$  the shear strains,  $\sigma_{ii}$  the normal stresses and  $\tau_{ij}$  the shear stresses,  $E_{ii}$  the moduli of elasticity,  $G_{ij}$  the shear moduli and  $\mu_{ij}$  the Poisson's ratios. Thereby, the first index signifies the strain direction and the second index the stress direction.

Selected property parameters of hardwood have been published by Kollmann (1951), Bodig and Jayne (1993), Szalai (1994), Pozgaj et al. (1997), Sell (1997), Wagenführ (2007), Kurjatko (2010) and Ross (2011), amongst others. However complete data sets for the three main wood directions that are sufficient for static calculations and modulations in wood construction and also for calculations on multi-layered boards, parquet or musical instruments with the finite element method rarely exist. Mostly. investigations on tension, compression and bending are carried out only parallel to the grain and the parameters for the other main directions (R, T), as well as the influences of grain and ring angle are lacking. Equally, the influence of the load type (tension, compression, bending) and of MC on the elastic constants and the Poisson's ratio are scarcely investigated. Also for the rheological characteristics (creep, relaxation, mechano-sorptive effects), the parameters, for the most part, are lacking. Certainly, Hering (2011) conducted detailed investigations on beech wood. For softwood, most investigations were carried out on Norway spruce since it is the most commonly used wood for construction (e.g. Neuhaus 1981).

Therefore, the aim of this work is to generate a preferable complete dataset of the mechanical properties of sycamore maple and beech under tension.

# MATERIAL AND METHODS

# Material

All specimens for the determination of the physical and mechanical properties were cut from logs of

- ➤ a sycamore tree (*Acer pseudoplatanus* L.) from Switzerland with a mean normal density of 626 kg/m<sup>3</sup> (at a MC of about 12%)
- A beech tree (*Fagus sylvatica* L.) from Switzerland with a mean normal density of 661 kg/m<sup>3</sup> (at a MC of about 12%)

The following mechanical properties were tested at the climates 20/35 (20°C and 35% relative humidity (RH)), 20/65, 20/85 and 20/95:

- Tensile strength, MOE and Poisson ratio under tension
- Fracture toughness K<sub>IC</sub> (mode I)

# Methods

# **Determination of the mechanical properties**

Tensile strength

Tensile strength was determined parallel to the grain according to DIN 52187:1979-05. Perpendicular to the grain tensile strength determinations were made using 95 mm long dog-bone-shaped specimens (cross-sectional area: max. 28 mm x 28 mm, min. 14 mm x 14 mm) according to Hering et al. (2012). 13-16 specimens were tested per direction and climate.

# Fracture toughness K<sub>IC</sub> (mode I)

Fracture toughness  $K_{IC}$  was determined according to DIN EN ISO 12737:2011-04 on compact specimens at RL, TL, RT and TR directions (first index = direction normal to the crack plane, second index = direction of crack propagation). 5-14 specimens were tested per direction and climate.

The static tests were carried out with a Zwick Z010 universal testing machine for tension perpendicular to the grain as well as fracture toughness, and a Zwick Z100 machine for tension parallel and fracture toughness.

## **RESULTS AND DISCUSSION**

#### Tensile tests

Table 1 shows the test results with increasing EMC (equilibrium moisture content), decreasing MOE and tensile strength. All values are reduced with increasing MC by about one-third (Fig. 1). For the wood directions at normal climate, the strength ratio of L, R, T is for tension 13 : 1.8 : 1 (for maple) and 14 : 1.8 : 1 (for beech).

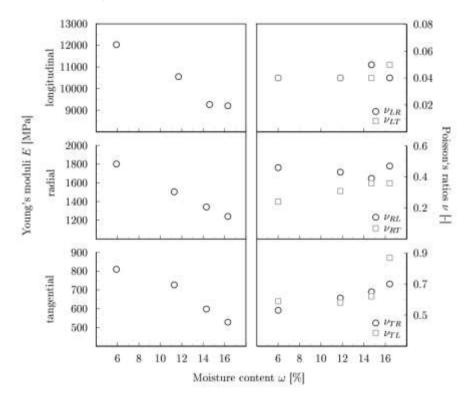


Fig. 1: Moisture-dependent Young's moduli and Poisson's ratios of European beech (Ozyhar (2012))

Table 1: Mechanical properties of European beech and sycamore maple at climate 20/65;  $\sigma$  = strength in tension (t), bending (b) and compression (c), MOE = modulus of elasticity,  $\tau$  = shear strength, w = impact bending strength,  $K_{IC}$  = critical stress intensity factor, L = longitudinal, R = radial, T = tangential,  $\rho$  = density, V = coefficient of variation.

Property	Direction	ρ [kg/m <sup>3</sup> ]	Mean value	V [%]
Troperty	Direction	maple	Witchi value	v [ /0]
ENT/ 21	т	-	110.4	20.1
$\sigma_t [N/mm^2]$	L	633	112.4	20.1
	R	630	16.2	8.8
	Т	630	8.9	7.2
MOE <sub>t</sub> [N/mm <sup>2</sup> ]	L	633	11450	23.5
	R	630	1205	10.0
	Т	630	688	3.8
K <sub>IC</sub> [MPa·m <sup>0.5</sup> ]	RL	670	1.08	18.3
	TL	620	0.70	11.4
	RT	690	0.91	10.7
	TR	589	0.54	17.0
		beech		
$\sigma_t [N/mm^2]$	L	661	96.7	28.4
	R	668	19.5	15.3
	Т	654	14.7	6.6
MOE <sub>t</sub> [N/mm <sup>2</sup> ]	L	661	10560	12.5
	R	668	1510	8.1
	Т	654	730	10.3
K <sub>IC</sub> [MPa·m <sup>0.5</sup> ]	RL	653	0.795	17.8
	TL	627	0.358	14.3
	RT	641	0.619	11.3
	TR	638	0.401	14.4

The reduction of MOE with increasing MC is similar to the reduction in strength.

## Poisson's ratio

Table 2 shows the Poisson's ratio ( $\mu$ ) at normal climate (20/65) determined from the tension and compression tests. The mean Poisson's ratios from tension and compression over all climates coincide quite well with values determined by Stamer and Sieglerschmidt in Kollmann (1951) for sycamore maple and with values of Bodig and Jayne (1993) for hardwood. The influence of MC on Poisson's ratio is not uniform so that the values increase or decrease depending on the direction (see Fig.1). These are the same results that we obtained for beech and maple. In contrast, Hering et al. (2012) observed a decrease of Poisson's ratio for beech with increasing MC in all directions.

<i>V</i> = coefficient of variation							
	maple		beech				
Direction	μ	V	μ	V			
	[-]	[%]	[-]	[%]			
RL	0.489	15.3	0.43	17.1			
TL	-	-	0.58	-			
TR	0.646	4.3	0.61	6.7			
LR	0.059	36.1	0.04	42.9			
RT	0.378	9.7	0.36	8.7			
LT	0.043	29.8	0.04	41.2			

Table 2: Poisson's ratio ( $\mu$ ) at normal climate (20/65) for tension

## Fracture toughness

Table 1 shows the fracture toughness  $K_{IC}$  of the different load directions at climate 20/65. A small difference between beech and maple is observed, with the values of maple being higher than the values of beech. The values are very high when compared with other wood species, like oak, and also higher compared to sugar maple (Stanzl-Tschegg et al. 2011) and are similar to ash in the RL and TL directions (Reiterer et al. 2002). Tests in the RL and RT directions result in clearly higher values compared to the TL and TR directions, which can be attributed to the influence of the rays. The fracture toughness within a crack plane was higher for crack propagation in the fibre direction than perpendicular to the grain, which is in contrast to the behaviour of different soft woods (Stanzl-Tschegg et al. 2011). All  $K_{IC}$  values are highly influenced by MC, with the greatest percentage decrease in the RL and TL directions (Fig. 2). In contrast, Logemann and Schelling (1992) found only a low MC influence for spruce in the TL direction.

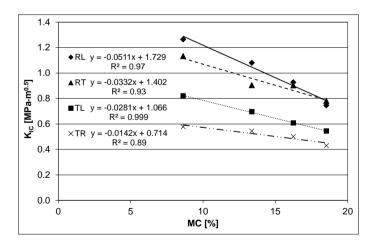


Figure 2: Fracture toughness  $(K_{IC})$  of maple dependent on moisture content (MC).

# CONCLUSIONS

A data set of mechanical properties in the three main directions was established for selected properties of European beech and sycamore maple wood. Until now, such complex data sets for hardwoods (ash, oak, beech) are rare, despite becoming more and more important in parallel to the increasing silvicultural availability of these species. The data set allows, within the elastic range, the calculation and simulation of multi-layered and three-dimensional wood structures with finite element methods. Still, investigations have to be carried out to determine the physical (sorption, swelling, diffusion, thermal conductivity) and rheological properties and the mechano-sorptive behaviour. Equally, further research is needed to analyse the plastic behaviour, which is particularly important in regard to compression perpendicular to the fibre.

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# LITERATURE

- Bodig, J., Jayne, B. (1993) Mechanics of Wood and wood composites. 2nd edition, Krieger Publishing, Malabar, Florida
- Brändli, U.-B. (ed.) (2010) Schweizerisches Landesforstinventar. Ergebnisse der dritten Erhebung 2004–2006. Birmensdorf, Eidgenössische Forschungsanstalt für Wald, Schnee und Landschaft WSL. Bern, Bundesamt für Umwelt, BAFU. 312 S.

Conners, T.E., Medvecz, P.J. (1992) Wood as a bimodular material. Wood Fiber Sci 24(4):413-423

- Hering, S. (2011) Charakterisierung und Modellierung der Materialeigenschaften von Rotbuchenholz zur Simulation von Holzverklebungen. Dissertation, ETH Zürich
- Hering, S., Keunecke, D., Niemz, P. (2012) Moisture-dependent orthotropic elasticity of beech wood. Wood Sci Technol (first online), DOI 10.1007/s00226-011-0449-4

- Keunecke, D., Sonderegger, W., Pereteanu, K., Lüthi, T., Niemz, P. (2007) Determination of Young's and shear moduli of common yew and Norway spruce by means of ultrasonic waves. Wood Sci Technol 41(4):309-327
- Keunecke, D., Hering, S., Niemz, P. (2008) Three-dimensional elastic behaviour of common yew and Norway spruce. Wood Sci Technol 42(8):633-647.
- Kollmann, F. (1951) Technologie des Holzes und der Holzwerkstoffe. 2nd edn., vol 1. Springer, Berlin
- Krackler, V., Keunecke, D., Niemz, P. (2010) Verarbeitung und Verwendungsmöglichkeiten von Laubholz und Laubholzresten. ETH Zürich, Eigenverlag, 151 S.
- Kurjatko, S. (2010) Parametre kvaliy dreva urcujuce jeho finalne pouzitie. TU Zvolen, 351 S.
- Logemann, M., Schelling, W. (1992) Die Bruchzähigkeit der Fichte und ihre wesentlichen Einflussparameter. Untersuchungen in Mode-I. Holz Roh-Werkst 50(2):47-52
- Neuhaus, F.H. (1981) Elastizitätszahlen von Fichtenholz in Abhängigkeit von der Holzfeuchtigkeit. Dissertation, Ruhr-Universität Bochum, 162 S.
- Ozyhar, T.; Hering, S.; Niemz, P. (2012) Moisture-dependent elastic and strength anisotropy of European beech wood in tension. Journal of Material Science , 2012 DOI: 10.1007/s10853-012-6534-8
- Pozgaj, A., Chonavec, D., Kurjatko, S., Babiak, M. (1997) Struktura a vlastnosti dreva. 2nd edn. Priroda, Bratislava, 485 S.
- Reiterer, A., Sinn, G., Stanzl-Tschegg, S.E., (2002) Fracture characteristics of different wood species under mode I loading perpendicular to the grain. Mater Sci Eng A 332:29-36
- Ross, R.J. (ed.) (2011) Wood Handbook. 2010 edition. Forest Products Society, Madison WI

- Schmidt, J. (2008) Modellierung und numerische Analyse von Strukturen aus Holz. Habilitation, TU Dresden
- Sell, J. (1997) Eigenschaften und Kenngrössen von Holzarten. 4th edn. Baufachverlag, Dietikon, 87 S.
- Sonnabend, J. (1989) Das Holz des Ahorns und seine heutige Verwendung. Parts 1-2. Holz-Zentralblatt 115:1680, 1683, 2020, 2024
- Sonnabend, J. (1990) Das Holz des Ahorns und seine heutige Verwendung. Part 3. Holz-Zentralblatt 116:26-27
- Stanzl-Tschegg, S.E., Keunecke, D., Tschegg, E.K. (2011) Fracture tolerance of reaction wood (yew and spruce wood in the TR crack propagation system). Journal of the Mechanical Behavior of Biomedical Materials 4:688-698
- Szalai, J. (1994) A faanyag és faalapú anyagok anizotrop rugalmasság- és szilárdságtana (Book title in English: Anisotropic strength and elasticity of wood and wood based composites), University of West Hungary, Sopron [in Hungarian]

Wagenführ, R. (2007) Holzatlas. 6th edn. Fachbuchverlag, Leipzig

Wedel, K. von (1964) Untersuchungen über Eigenschaften, Verwertung und Verwendung des Ahornholzes. Dissertation, Georg-August-Universität Göttingen, Hann. Münden, 176 S.

# Surface modification of Oakwood with Tricine

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Keywords: Oak wood, modification, Tricine, hardness

## ABSTRACT

This study seeks to assess the potential use of a surface treatment for oak wood by a chemical modification. For the experiments of this study, the alpha-amino acid Tricine was used, which is commonly used as a buffer. Tricine is a harmless substance from which no environmental threat emanates. Since Tricine is a zwitterion with only low reactivity at room temperature, higher temperatures for the treatment should be used. So it should be possible that the functional groups of Tricine form associations with wood components. It is known from the scientific literature that extractives as well as cellulose and lignin can react with amino acids. Accordingly a variety of reactions that can have an influence on wood properties should proceed by an impregnation of amino acids in wood. The oak wood was modified with Tricine through a full impregnation and with a superficial application. The hardness and the swelling properties of the modified lamellae were then tested. The tested lamella, both the coated and the impregnated specimen showed a significant increased hardness compared to the reference. The impregnated oak wood had the highest tested hardness with an average of 53 % increase. The hardness of the coated lamellae increased in average by 31 %. Compared to conventional coatings, or other chemical modifications of solid wood, similar values for an increase in hardness can be achieved. The surface treatment with Tricine was found to be a valuable alternative to the impregnation process, since energy and chemicals can be saved and the subsequent drying is simplified. Disadvantages of the coating, however, are the not improved swelling properties which occur due to the impregnation process.

# INTRODUCTION

The modification of solid wood is often the chosen way to improve wood properties. Known methods from the literature have the primary aim to improve the durability as well as the dimensional stability of wood. This is different for the modification with Tricine. The intention is rather the improvement of the hardness characteristics for indoor applications. It is not assumed that a treatment with Tricine increases the durability, because the substance has no biocidal properties and the water absorption is only slightly influenced. In a first and until now unpublished study by Hauptman et al. (2012) it could be shown that due to modification with Tricine results in a bulking effect and leads to an increased dimensional stability.

Basically, it is known from the scientific literature that extractable wood components, e.g. Tannins from oakwood, react with proteins or amino acids and polymerize (Gustavson 1956). It is also conceivable, and partly known that amino acids can react with further wood components, e.g. with the structural component cellulose (Cateto and Ragauskas 2011)(Fig.1) Also the reaction of lignin with amino acids can be found in the literature (Kawamoto et al. 1992). In the study by Hauptmann et al. (2012) various reactions of amino acids with wood components were detected. The reactions resulted in a significant increase in the hardness of wood. Also a decrease in tensile strength could be identified. The chemical reactions led to a change in the cellulose flexibility interferes with the strength of the cellulose chains. So the reduction of the tensile strength can be explained.

The hardness increase achieved by a Tricine modification is quite comparable to other chemical modifications of solid wood. It is known from the literature that modifications with silane, DMDHEU or melamine formaldehyde resins achieve increases of 30-74 % (Dieste et al. 2008, Donath 2005, Gindl et al. 2004). It should be noted however that this hardness increases are due to the filling of the cell lumen and not on the chemical modification itself. However, this can be assumed with a Tricine modification, because only very low WPG values are reached (Hauptmann et al. 2012).

In this study it should be determined if a simple coating of Tricine solution can increase the hardness of solid oak wood. The process of impregnation is costly and should be avoided also because of the further drying process of the treated wood. A surface modification is easier to apply and could also save a significant amount of chemicals.

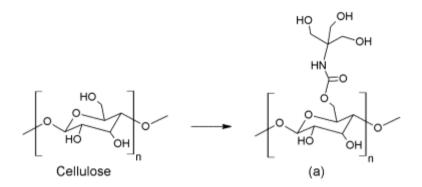


Figure 1: Schematic reaction of cellulose with Tricine (a)

## **EXPERIMENTAL METHODS**

For the investigations six oak lamellae with the dimensions 12x7x0.5 cm were coated with a 15 % Tricine solution (80-100 g/m<sup>2</sup>). For comparison, six oak lamellae with the same dimensions were impregnated in a surplus process with a 15 % Tricine solution. The impregnation process was carried out for 6 h with 6.5 bar over pressure. Both the coated and the impregnated samples were then cured in a temperature program. The samples were heated in this case, for one day each with 40°, 60° and 103°C. For reference, six oak lamellae were impregnated only with water and exposed to the same temperature program.

The surface hardness of the treated and untreated samples was determined using a universal testing machine (Zwick 020) with a steel ball indenter (Brinell) of 10 mm diameter. The resistance of indentation determined the hardness, according to EN 1534. The wood samples were measured at 23°C and 65 % relative humidity. Four measurements on each wood sample were performed.

To determine the swelling properties of the coated an impregnated oak wood, the lamella were impregnated with water until maximum water absorption. Also the reference samples were impregnated with water for comparison. The samples were measured before and after the water treatment in length, height and width direction.

#### **RESULTS AND DISCUSSION**

The tested lamella, both the coated and the impregnated specimen showed a significant increased hardness (Anova, p=0.00) compared to the reference

(Fig. 2). Also the impregnated lamellae showed a significant increase in hardness (p=0.00) compared to the coated lamellae. However, the experiment confirmed the hypothesis that only a Tricine coated surface should be enough to increase the hardness. An impregnation and the modification of the complete wood excel the coating because of the following reason. The thin layer of the coated modificated wood is not deep enough to prevent the perpetuation and the transferrin of the force into the unmodificated and softer areas of the oak wood. A mixed hardness value gets created and decreased the overall hardness value.

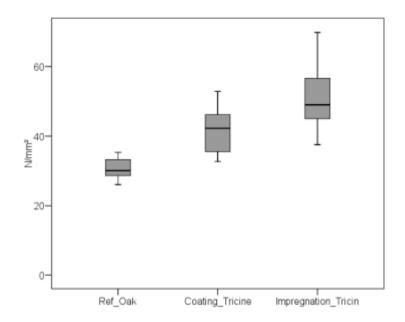


Figure 2: Hardness of the coated and impregnated oak wood with Tricine

Compared to conventional coating systems, the results of the Tricine treated samples are showing similar values. The Brinell hardness of coated wood with a Cellulosic or Polyurethane varnish is increased by 22 % - 40 % (Kurt and Özcifci 2009). The Tricine impregnated samples exceed these values and achieve an increased hardness of 53 % in average.

The obtained hardness improvements are also comparable with several modification processes. However, those are not designed for a pure increase in hardness but rather on the durability of treated timber.

Nevertheless, an increased hardness is seen as beneficial (Gindl et al. 2004, Trinh et al. 2012). Modified wood products, with N-methylol melamine, melamine formaldehyde or siloxanes achieve hardness increases from 33 %-53 % (Gindl et al. 2004, Trinh et al. 2012, Gosh 2010). Through a complete filling of the cell lumen a clearly higher hardness can be achieved. By impregnating with wax, hardness increases of 500 % were realised (Scholz 2011).

In general the advantages of a surface treatment are obvious. Due to the energy-saving application of the Tricine solution, the simplified drying process and yet an increased hardness, the coating process leads to significant advantages. An advantage of the impregnation is not only a higher hardness but also reduced swelling properties. Due to changes in the cellulose structure, the water absorption is lower compared to the reference. This does not apply for the coated wood, since the modified part is much smaller.

Table 1: Aver	Table 1: Average volume swelling of the coated and the impregnated wood samples						
Samples	∆ Length [%]	∆ Height [%]	∆ Width [%]	∆ Volume [%]			
Reference	0.7	2.1	5.5	8.8			
Coated	0.3	1.5	7.6	9.9			
Impregnated	0.9	0.7	3.0	4.7			

The average volume swelling is shown in Tab.1 and it becomes apparent that the swelling properties of the coated samples are similar to the reference. The general sorption properties of the modified coated wood should differ only slightly from the reference.

# CONCLUSION

An easy to apply and harmless process could be found, that can increase the hardness of wood. By only coating the oak wood with a 15% Tricine solution a significant increase in hardness can be recognized with also a minimum of increased swelling properties because of chanced chemical structures over the entire cross-section of the wood.

# REFERENCES

CATEGO, A.C. AND RAGAUSKAS, A. (2011) Amino acid modified cellulose whiskers. *RSC Advances*, **9**(1), 1695-1697.

DIESTE, A., KRAUSE, A., BOLLMUS, S. AND MILITZ, H. (2008) Physical and mechanical properties of plywood produced with 1.3-dimethylol-4.5-dihydroxyethyleneurea (DMDHEU)-modified veneers of Betula sp. and Fagus sylvatica. *Holz als Roh- und Werkstoff.* 66(4), 281-287.

DONATH, S. (2005) Treatment of woods with silans. Sierke Verlag.

EUROPEAN STANDARD EN 1534 (2011) Wood flooring - Determination of resistance to indentation (Brinell) - Test method

GINDL, W., HANSMANN, C., GIERLINGER, N., SCHWANNINGER, M., HINTERSTOISSER, B. AND JERONIMIDIS, G. (2004) Using a water-soluble melamin-formaldehyde resin to improve the hardness of Norway spruce wood. *Journal of Applied Polymer Science* **93**(4), 1900-1907.

GOSH, S.C. (2010) Wood modification with functionalized polydimethylsiloxanes. *Sierke Verlag*.

GUSTAVSON, K.H. (1956) The Chemistry of Tanning Processes. *New York: Academic Press Inc.* 162-201.

Hauptmann, M., Gindl-Altmutter, W., Liebner, F., D'Amico S. and Hansmann, C. (2012) Wood modification with Tricine. *Unpublished Paper*.

KAWAMOTO, H., NAKATSUBO, F. AND MURAKAMI, K. (1992) Protein adsorption capacities of lignin samples. *Mukuzai Gakkaishi*. **38**(1), 81-84.

KURT, S. AND OZCIFICI, A. (2009) Effect of various fire retardants on brinell hardness of some wood. *Bioresources*, **4**(3), 960-969.

SCHOLZ, G. (2011) Veredelung von Massivholz mit heißschmelzenden Wachsen. Cuvillier Verlag.

TRINH, H.M., MILITZ, H. AND MAI, C. (2012) Modification of beech veneers with N-methylol-melamine compounds for the production of plywood. *European Journal of Wood and Wood Products.* **70**(1), 421-432.

# Superposition of thermal- and photodegradation for wood monitored by colour measurement\*

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Keywords: Wood, Photodegradation, Thermal degradation, Colour change

# ABSTRACT

When we would like to get the best usability from a material first we need to know the properties of the material completely. The aim of this research was the best cognition of wood as a raw-material, more exactly the photodegradation of the solid wood at elevated temperature. This research analyses the effect of the photodegradation on behalf of the colour changes.

In order to see what kind of effect has the temperature on colour change we have irradiated the solid wood samples with mercury vapour lamp, first at 30°C and than at 80°C. The results showed, that higher temperature (80°C) created greater redness change than the lower temperature (30°C) did. We experienced also that the yellowness has not changed significantly at higher temperature. Pine samples showed 57% higher redness change at 80°C than at 30°C,. For spruce, ash and poplar samples these data were 33%, 40% and 15%, respectively.

The results of this research shows us, that the extractive content plays a very important role in photodegradation at elevated temperature.

# INTRODUCTION

The wood is one of the most beautiful materials created by the nature. Its mechanical skills and unbelievable multiple colour diversity makes the wood a perfect material to work with. The wood has a unique structure which is composed from cellulose biopolymer and lignin naturally arranged into tubular structures and from a cylindrically layered composite.

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The colour inhomogeneity of wood is one of the most beautiful colour harmony created by the nature (L. Kucera, S. Katuscak, 1992). We – human kind – feel an atavistic leaning to the warmness of the colour of the wood, because there is the red and the yellow the two most dominative hue of colour of wood.

The cellular structure of wood has a lot of micro mirrors aligned parallel to the grain. Gloss from these micro mirrors gives us more elegant, soft, natural and beautiful texture than that of plastics and metal (M. Masuda, 2001).

To maintain this perfect colour we have to be careful because the colour of solid wood is really sensitive to light and heat. The main factor that causes the greatest changes in the colour of wood is sunlight during outdoor exposure (L. Tolvaj, K. Mitsui, 2005). The surface starts yellowing and turns grey (L. Tolvaj, G. Papp, 1999). Chemical analyses show that the deterioration is primarily related to the decomposition of lignin (U. Müller, M. Rätzsch, M. Schwanninger, M. Steiner, H. Zöbl, 2003). The chromophoric groups of lignin are strong ultraviolet (UV) light absorbers. The energy of absorbed UV photons is large enough to create free phenoxyl radicals. These free radicals react with oxygen to produce carbonyl chromophoric groups (K.K. Pandey, 2005). The chromophoric groups are then responsible for the colour change of wood.

Our investigation's main purpose was to analyse the impact of the elevated temperature to the colour modification of the wood.

Because we realised that the thermal effect is exponentially proportional to the temperature, we submit our samples to the heat at two different temperatures i.e.,  $30^{\circ}$ C and  $80^{\circ}$ C.

# MATERIALS AND METHODS

During the research we examined 2 hardwood and 2 softwood species, which were ash (*Fraximus excelsior* L.) and poplar (*P.Xeuramericana Pannonia*), Scots pine (*Pinus silvestris* L.) and spruce (*Picea abies* Karst.). Two pieces of samples were cut 100x30x10 (mm) from the same wood board, and ten places were marked for the colour measurement on all of the samples.

Because we made 10 measures in each samples of species, we have got 20 results per species.

As mentioned two different air temperatures - 30°C and 80°C - were used in the irradiation chamber. To irradiate our samples we used a mercury vapour lamp which emits strong UV light. We have put the samples 64 cm from the lamp, and the total electric power what we used were 800 W. Total irradiation time was 200 hours. The exposures were interrupted after 8; 20;

40 and 90 hours because we need to measure the change of the colours. We also measured the colour change before and after the irradiation. Measurements were carried out with a colorimeter (Konica-Minolta 2600d). The L\*, a\*, b\* colour co-ordinates were calculated based on the  $D_{65}$  illuminant and 10° standard observer with a test-window diameter of 8 mm. A series of samples were treated in the same chamber set for 80°C but without light irradiation. This way we were able to observe that the effect of pure thermal degradation was determined.

# **RESULTS AND DISCUSSION**

During the investigation we realised that in the first 20 hours of light irradiation the lightness decreased really fast. After this period, the lightness change was moderate and almost linear.

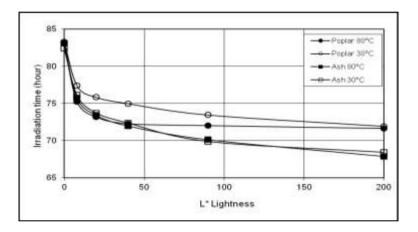


Figure 1: The lightness change of poplar and ash samples at 30°C and at 80°C temperatures

During the first quarter of the irradiation we concluded that the irradiation at 80°C caused slightly greater lightness decrease than at 30°C. The only exception was the poplar, which showed a considerably greater lightness decrease at the higher temperature.

Fig. 2 represent the lightness change of pine and sruce samples caused by photo-irradiation at 30°C and at 80°C temperatures.

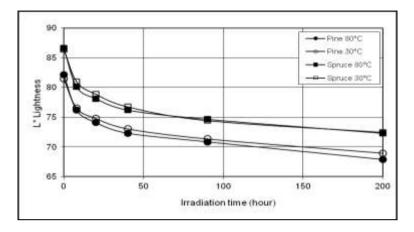


Figure 2: The lightness change of pine and spruce samples at 30°C and at 80°C temperatures

The greatest difference between the two types of irradiation was observed in red colour change, which we can see on the figure 3 and 4.

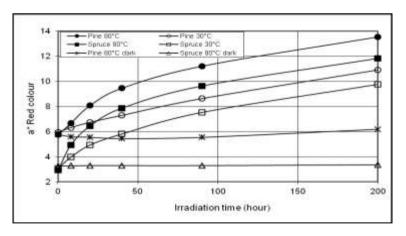


Figure 3: Red colour change in case of pine and spruce samples

The red colour change increased during the process continuously. At the very beginning of the investigation (in the first 8 hours) we found that the increase was more intensive for hardwood than for softwood.

We also saw clear that the light irradiation at 80°C produced considerably greater redness than the light irradiation at 30°C in all cases. The pure thermal treatment at 80°C in total darkness produced negligible redness increase, as it is presented in Figure 3 and figure 4.

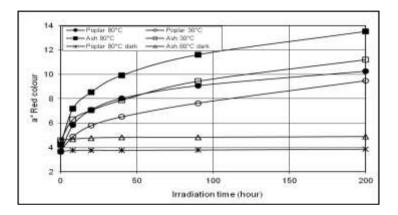


Figure 4: Red colour change in case of ash and poplar samples

It is clear, that the redness increase of light irradiation at 80°C is not the superposition of the effect of the light irradiation at 30°C and the effect of thermal treatment at 80°C in total darkness. The elevated temperature amplifies the consequence of light irradiation. Pine samples showed 57% higher redness change at 80°C than at 30°C during the 200 hours light exposure. The same percentages for spruce, ash and poplar are 33%, 40% and 15%, respectively. The chromophoric groups in wood are located in the lignin, in the extractives and in their derivatives. Lignin derivatives play main role mostly in yellowing. In the case of poplar wood the low extractive content could be the reason for the low thermal effect during light irradiation. This result shows the importance of extractives in the redness change. Based on experiences the indoor wooden constructions change their colour towards brown over the years. Probably, the amplification effect at elevated temperature is a contributing factor to the photodegradation, that responsible for that kind of colour changes. The thermal effects are exponentially proportional to the temperature. That is why the redness change needs long time period at room temperature.

Yellowing is the main colour alteration effected by the photodegradation. Yellowing is produced mainly by the degradation of lignin. The change of the yellow colour co-ordinate is presented in figure 5 and 6.

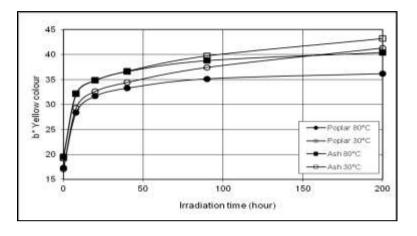


Figure 5: Yellow colour change in case of poplar and ash samples

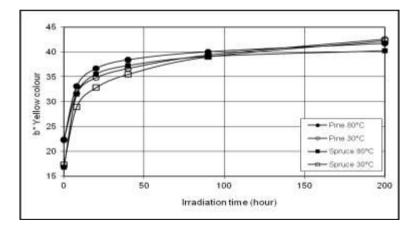


Figure 6: Yellow colour change in case of poplar and ash samples

We can also see on the figures, rapid yellowing happens during the first 20 hours of treatments; followed by a moderate but continuous yellowing. Conifers presented a little greater yellowing at 80°C than at 30°C up to 90 hours treatment. In contrast, hardwood suffered greater yellowing at 30°C

than at  $80^{\circ}$ C (ash showed only in the second part of irradiation). It is also really interesting that the yellowing was not altered by the thermal effect. After all, we have to assume that we need further investigations regarding to the chemical background of the yellowing.

# CONCLUSIONS

In this study we found out that the temperature has a considerable effect on the photodegradation of the wood.

We were able to prove that the higher temperature ( $80^{\circ}$ C) has greater effect on the redness increase that the lower ( $30^{\circ}$ C) temperature. The most considerable changes were made in pine samples – this sample showed 57% higher redness change at  $80^{\circ}$ C during the 200 hours than in  $30^{\circ}$ C –. Also the other samples showed higher redness changes on the higher temperature (spruce, ash and poplar were 33%, 40% and 15%,)

As we supposed the extractive content has a significant role in thermal discolouration during photodegradation.

# REFERENCES

L. KUCERA, S. KATUSCAK, Das Phenomen Holzfarbe. Holz-Farbe-Gestaltung, 24. Fortsbildungkurse der Schweizerishen Arbeits-gemeinschaft für Holzforschung (SAH) in Weinfelden, (4-5 November) Zürich (1992) 43-52.

M. MASUDA, *Why human loves wood grain figure?* Extraction of vision-physical characteristics deeply related to impression, ICWSF Conference (5-7 Sept.) Ljubljana (2001) 11-23.

K. MITSUI, Changes in the properties of light-irradiated wood with heat treatment. Part 2. Effect of light-irradiation time and wavelength, Holz Roh Werkstoff 62 (2004) 23-30.

K. MITSUI, A. MURATA, S. TSUCHIKAWA, M. KOHARA, Wood Photography Using Light Irradiation and Heat Treatment, Color Res. Appl. 29 (2004) 312-316.

K. MITSUI, L. TOLVAJ, G. PAPP, J. BOHUS, S. SZATMÁRI, O. BERKESI, Changes in the properties of light-irradiated wood with heat treatment. Part 4. Application of laser, Wood Res. Slov. 50 (2005) 1-8.

U. MÜLLER, M. RÄTZSCH, M. SCHWANNINGER, M. STEINER, H. ZÖBL, Yellowing and IR-changes of spruce

L. TOLVAJ, D. VARGA, R. NEMETH, S. MOLNAR, Color modification of black locust depending on the steaming parameters, Wood Res. Slov. 55 (2010) 81-88.

wood as result of UV-irradiation, J. Photochem. Photobiol. B: Biol. 69 (2003) 97-105.

L. TOLVAJ, G. PAPP, Outdoor Weathering of Impregnated and Steamed Black Locust,

ICWSF'99 Conference, (14-16 July) Missenden Abbey (UK) (1999) 112-115.

L. TOLVAJ, K. MITSUI, Light Source Dependence of the Photodegradation of Wood, J. Wood Science 51 (2005) 468-473.

L. Tolvaj, L. Persze, L. Albert, Thermal degradation of wood during photodegradation, J. Photochem. Photobiol. B: Biol. (2011) doi: 1016/j.jphotobiol.2011.07.005

K.K. PANDEY, Study of the effect of photo-irradiation on the surface chemistry of wood, Polym. Degrad. Stab. 90 (2005) 9-20.

# Feature of contact angle of ageing Beech and Birch surfaces\*

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Keywords: surface tension, contact angle, ageing, Xenon radiation,

#### ABSTRACT

Surface tension or surface free energy of wood surfaces is one of the most important attributes of gluing and coating wood, both with natural and synthetic resin. The state of the adherent surfaces is critical to the achievement of the necessary adhesion. According to the Young-Dupré equation the higher the surface tension of a solid the better its wetting is. Freshly cut surfaces were supposed to have higher surface tension than those cut for prolonged time and their ageing process was monitored. In order to get a systematic approach and comparable results ageing of wood surfaces was induced by artificial Xenon radiation, simulating indoor conditions. Samples of two wood species Beech (*Fagus Sylvatica L.*) and Birch (*Betula pendula*) were prepared by planning and sanding and were investigated during 40 hours exposure to Xenon radiation. Up to 15 hours there was an increase in the contact angle followed by a decrease during the last 25 hours of radiation.

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<sup>\*</sup> This (research) was supported by the European Union and co-financed by the European Social Fund in frame of the project "Talentum - Development of the complex condition framework for nursing talented students at the University of West Hungary", project ID: TÁMOP 4.2.2.B-10/1-2010-0018

The feature of surface tension during ageing under artificial Xenon radiation follows an exponential function of time for both planed Beech and Birch surfaces and was described with a logarithm natural function of time for sanded Beech and Birch surfaces.

# INTRODUCTION

Surface tension of solid wood is the main attribute of sufficient wetting and good adhesion of fluids like adhesives and lacquers. Wetting is good when the contact angle becomes very small, or disappears (River et al., 1991). A freshly prepared wood surface assures the highest adhesion

(Sernek, 2008). M. Sernek et al. investigated the wettability and adhesion of reactivated southern pine surfaces. Improvement in adhesion due to surface chemical treatment was not evident for all specimens, the choice of the adhesive drastically impacted the adhesion of bonded assemblies.

(Mantanis et al.1997) investigated the contribution of thermodynamic work of adhesion and contact angle to the wettability and surface tension of Sitka Spruce and Douglas -fir. They found that 75-80% of the total surface free energy was attributed to the dispersion forces. Gindl et al. described an increase of carbon atom composition with 6% after 7 days ageing in natural conditions and in the same time a decrease with 6% of oxygen atom composition on the microtomed beech surfaces. M. de Meijer conducted a comparison of surface energy determination methods of spruce and meranti. Measurements of acid and base parameters of wood surfaces seemed not to be very reliable because of its strong dependence on the measuring conditions. It was noted that thermodynamic equilibrium conditions assumed by Young's equation are generally not fulfilled with wood surfaces because of chemical heterogeneity, surface roughness and the absorption of the test solvent. Based on the upper considerations during our tests the testing time was set at 1 second after release of the drop, and two diffuse porous wood species were chosen to reduce the influence of the anatomical heterogeneity to the minimum. Wood has an inhomogeneous, anisotropic structure, and there are relevant differences in microstructure of the different wood species. Besides the differences in the anatomical microstructure sanding and planning as machining techniques also influence the physico-chemical properties of the surface achieved. Two diffuse-porous, relatively homogeneous wood species were chosen for the tests in order to minimize the possible influence of some local structural elements like large pores, relevant structural differences in early wood and late wood.

As a natural material, wood is also subject to degradation. Investigations were conducted in order to describe the time dependent behavior of planed

and sanded Beech and Birch surfaces due to sun radiation for a better understanding of the differences or similarities of their surface ageing process. Instead the natural sun radiation which varies with the seasons and also during the day, an artificial Xenon lamp radiation was undertaken in order to get a systematic approach and comparable results. Between the potential artificial radiation types (mercury lamp, Xenon lamp) the Xenon light is able to simulate the sunlight more properly than mercury lamps light (Tolvaj, 2011). The two wood species were subjected to artificial Xenon radiation and their behavior was investigated, with attention on the influence of the type of machining too.

# **EXPERIMENTAL METHODS**

1) Sample preparation: 2 samples of 55 mm x 95 mm x 25 mm with planed surface and 2 samples of 55 mm x 95 mm x 25 mm with sanded surface were prepared both from Beech and Birch wood species (Molnar et al., 2002), all tangential cut sections. 10 measurements were performed on planed surfaces and 10 measurements on sanded surfaces after each artificial Xenon radiation cycle.

Half of the samples were planed with a four knife cutter-head thickness planer and half of the samples were sanded with a wide belt sanding machine (grit size 120) and cut to size by circular saw. All samples were conditioned 7 days at 22  $^{\circ}$ C and 65% relative temperature prior to starting Xenon radiation.

2) The apparatus of artificial radiation and the curing procedure: Radiation curing was performed using an artificial Xenon radiation apparatus Original Hanau Suntest. The apparatus is equipped with Xenon bulb having a sunlight spectra filter. The apparatus is equipped with Xenon bulb having sunlight spectra, due to a "Daylight" filter of  $0,51 \text{ W/m}^2$  irradiation intensity, with a UV peak at 340 nm. A cooling unit keeps the chamber temperature at  $38^{\circ}$ C. Changes in contact angle due to artificial xenon radiation were measured in 1-, 3-, 5-, 8-, 10-, 15-, 20-, 30- and 40 hour cycles systematic.

3) Measurement of contact angle: in contact angle measurement, a drop of liquid was placed on the surface of wood sample. It was assumed, that the liquid does not react with the solid. It was emphasized that contrary to ideal smooth surfaces, the drop of water is distorted along the grains, taking a form of a semi oval sphere.

The contact angle was measured as the angle between the outline tangent of the smaller diameter and surface (Fig. 1).

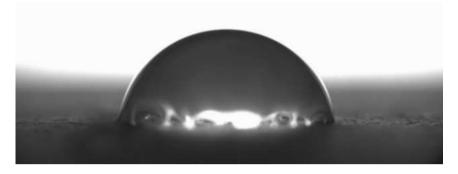


Figure 1: Drop shape taken along the smallest diameter of a semi oval sphere

For to perform and evaluate the measurements a computer aided PG-X goniometer as measuring instrument was used, with a measuring drop of 0,5  $\mu$ l, and the contact angle was automatically detected and measured at 1 sec after the release of the droplet, as previously agreed. As test liquid distilled water was used. Before each measurement the measuring instrument was calibrated. All measurements were performed in a laboratory conditioned to 65% RH and 22°C.

4) The Beech planed samples were of an average roughness of  $R_z$ = 25 µm and sanded samples were of  $R_z$ = 20 µm. Roughness was measured with a stylus tip instrument Perthen S3P, and  $R_z$  was found the most appropriate parameter to describe the status of the surface based on earlier studies (Csiha, 2004).

5) Results of contact angle measurement were processed in Microsoft Excel and Statistica programs and are presented in Table 1. Mathematical average was generated for each 10 batches of contact angle values of the same sample; evaluation was performed upon these data and the artificial ageing time/contact angle graphs.

	Beech				Birch			
	Sanded		Planed		Sanded		Planed	
	x	S	x	S	x	S	x	S
0h	24,77	3,37	54,75	4,87	19,18	5,06	54,32	9,11
1h	70,37	11,66	94,79	10,23	111,41	7,52	95,055	5,71
3h	98,925	3,96	95,67	4,54	116,195	6,23	96,26	6,2
5h	87,47	10,78	102,075	10,28	107,975	7,16	93,41	7,59
8h	100,3	6,99	92,25	5,43	113,07	3,57	102,91	6,35
10h	103,7	4,87	95,25	5,37	112,6	3,19	106,27	3,78
15h	102,7	4,06	96,99	5,08	108,1	3,18	101,87	7,85
20h	98,87	8,5	88,57	5,24	102,71	4,58	101,2895	4,53
30h	82,25	10,42	58,75	8,99	105,62	3,77	94,15	4,81
40h	71,95	10,35	55,55	9,19	103,4	3,94	88,6	4,97

Table 1. The average values of contact angle and its spread

The contact angle is linked to the surface energy and one can calculate the surface energy. Three parameters influence the shape of drop at wood surfaces: the wood/liquid interfacial tension ( $\gamma_{lw}$ ), the wood/vapour interfacial tension ( $\gamma_{vw}$ ) and the liquid/vapor interfacial tension ( $\gamma_{lv}$ ). These three parameters are linked with the contact angle by the Young/Dupré equation. The PG-X goniometer can be adjusted to measure both the contact angle and the surface tension of the wood/vapor interface. We considered that the primary parameter of measurement is the contact angle. Since the two parameters  $\gamma_{lv}$  and  $\gamma_{lw}$  are considered constant, the measured contact angle directly shows the trend of  $\gamma_{vw}$ . An increasing contact angle means that the surface tension of the solid is decreasing. In the present study the development of surface tension was evaluated by means of contact angle.

#### **RESULTS AND DISCUSSION**

In case of the two investigated species and the two machining types, the contact angle was increasing in the first 15 hours of artificial Xenon radiation (Fig.2).

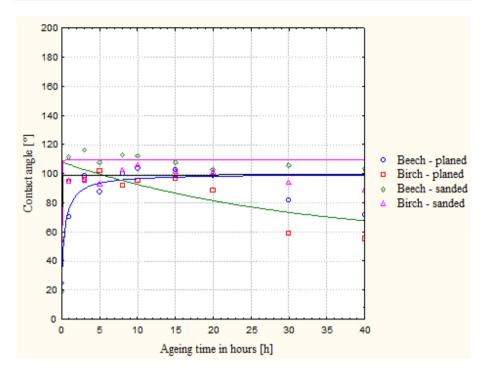


Figure 2: Development of contact angle during artificial Xenon radiation on Beech and Birch samples

There was a difference found between the feature of planed and sanded surfaces. The planed surfaces for both Beech and Birch samples were described as logarithm natural function of time (Eq. 1).

$$y = \frac{b^2}{x - b^1} + b^0$$
(1)

The sanded surfaces for both Beech and Birch samples were described as exponential function of time (Eq. 2).

$$y = b2 * (e^{-b1 * x} - e^{-b0 * x}) + b3$$
<sup>(2)</sup>

For the two wood species and the two machining types the character of the evaluation of the contact angle with the elapsed time of artificial Xenon radiation fallows the upper equations with determination coefficients as given in Table 2.

Table 2. The determination coefficient of the equations			
Determination coefficient of the logarithm natural and exponential equations $({\bf R}^2)$			
0,89			
0,94			
0,95			
0,99			
-			

Table 2 The determination coefficient of the as ...

For sanded Beech and Birch the tightness was found quite high, for planed Birch as well, but the planed Beech samples were presenting a somewhat lower  $R^2$ . The  $b_0$  parameter in the equations above for the different wood species and machining types has always the value of the first parameter measured at 0 hour Xenon radiation on the sanded and planed surfaces. The b coefficients are given in Table 3.

Table 3. The b0-b3 coefficients of the equations         The b0, b1, b2 and b3 coefficients						
	Beech - Planed	Beech - Sanded	Birch - Planed	Birch - Sanded		
b0	54,75	99,91	54,32	109,82		
b1	0,03	0,47	-0,51	0,01		
b2	55,08	36,07	44,32	0,26		
b3	52,92	-	54,32	-		

The contact angles of Birch samples both sanded and planed had lover spread during the artificial radiation than the Beech samples (Table 1). The sanded Birch samples had higher contact angles than the planed Birch samples, whilst the spread of the sanded Birch samples was lower than the spread of the planed ones. This means that sanding created a structure where the drop takes the same shape with high probability, whilst on the glossier planed surfaces the anatomical constituents (unevennesses) have a higher influence on the shape of the drop than machining.

#### CONCLUSIONS

There was a difference found between the feature of contact angle development under Xenon radiation of planed and sanded surfaces. The planed surfaces for both Beech and Birch samples were described as logarithm natural function of time whilst the sanded surfaces for both Beech and Birch samples were described as exponential function of time.

Machining proved to be a stronger influencing factor than the wood species. Planed surfaces had a lower contact angle than those sanded, meaning that the distilled water could spread easier on the planed surfaces than on those sanded with grit size 120. In case of the investigated two wood species considering the machining techniques also, the most relevant contact angle increase occurred during the first 15 hours of artificial Xenon radiation, followed by a decrease during the last 25 hours of radiation. It is supposed that the concentration of oxygen and carbon atoms on the surface also changed accordingly. Further investigations are planned to confirm the supposition.

# ACKNOWLEDGEMENTS

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#### REFERENCES

MOLNÁR S., BARISKA M., (2002) Wood Species of Hungary, *Szaktudás Kiadó Ház*, Budapest (70-78, 150-154, 178-176).

RIVER B. H., VICK C. B., GILLESPIE R. H. (1991) Wood as an adherend, Marcell Decker, Inc., Chapter 1 from Vol. 7 Treatise on Adhesion and Adhesives. p.: 89-90.

CS. CSIHA, (2004): Roughness measuring of wooden surfaces – new valuation criteria: Internationale Fachtagung Dresden 11-13 Mai 2004, Werkstoffe und Verfahren für Möbelfertigung und Innenausbau. p. 235-240.

TOLVAJ L., PERSZE L. "A napsugárzás mesterséges fényforrásokkal történő imitálásának problémája," *Faipar - Scientific Journal of Wood*, no. 2-3, 2011, pp. 19-26.

MEIJER M., HAEMERS S., COBBEN W., MILITZ H. (1999) Surface energy determinations of wood: a comparison of methods and wood species, Interactions between wood and coatings with low organic solvent content, Wageningen Universiteit. MANTANIS G. I., YOUNG R. A. (1997) Wetting of wood, Spinger-Verlag, Wood science and technology, 31, p 339-353.

GINDL M., REITERER A., SINN G., STANZL-TSCHEGG S. E., (2004) Effects of surface ageing on wettability, surface chemistry, and adhesion of wood, Spinger-Verlag, Holz Roh Werkst 62, p. 273-280.

- SERNEK M., GLASSER W. G., KAMKE F. A., (2008) Enhancing Bondline Performance –
- Final Conference of COST E34 Bonding of Timber, Sopron in 6-7 Mai 2008, Hungary

ROSS, R.J., DEGROOT, R.C. AND NELSON W.J. (1994) Technique for the non-destructive evaluation of biologically degraded wood. *Experimental Techniques*, **18**(5), 29-32.

# Computation of the specific heat capacity of frozen poplar wood during its defrosting

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**Keywords:** frozen wood, effective specific heat capacity, wood defrosting, bounded water, free water, computation

# ABSTRACT

An approach for the computation of the effective specific heat capacity  $c_{\rm we}$  of frozen wood during its defrosting has been suggested. The approach takes into account the physics of the process of thawing of the ice, which is created in the wood by both the hygroscopically bounded and the free water. It reflects for the first time also the influence of the fiber saturation point  $u_{\rm fsp}$  of the separate wood species on the value of their  $c_{\rm we}$  during wood defrosting and the influence of the temperature on the  $u_{\rm fsp}$  of frozen and non-frozen wood.

At temparatures lower than 271.15 K the value of  $c_{\rm we}$  has been presented as a sum from the specific heat capacity of the frozen wood itself  $c_{\rm w}$  and the specific heat capacity of the ice, which is formed by the freezing of the bounded water in the wood  $c_{\rm bw}$ . In the temperature range between 271.15 K and 272.15 K the value of  $c_{\rm we}$  has been presented as a sum from  $c_{\rm w}$  and the specific heat capacity of the ice, which is formed by the freezing of the free water in the wood  $c_{\rm fw}$ . At temperatures higher than 272.15 K the wood does not contain ice.

For the computation of  $c_w$ ,  $c_{bw}$ ,  $c_{fw}$ , and  $c_{we}$  according to the suggested approach a software program has been prepared, which has been input in the developed by Microsoft calculation environment of Visual Fortran Professional. With the help of the program computations have been made for the determination of  $c_{we}$  of often used in the plywood production poplar

wood with moisture content  $0.2 \le u \le 1.2$  kg.kg<sup>-1</sup> at the temperature range between -1 °C and -60 °C during defrosting of the wood.

The created mathematical description of  $c_{\rm we}$  has been input in the earlier suggested by the author non-stationary model of defrosting processes in cylindrical wood materials. The updated model has been solved with the help of explicit schemes of the finite difference method. The change in the transient temperature distribution in  $\frac{1}{4}$  of the longitudinal section of subjected to defrosting poplar frozen log is graphically presented and visualized with the help of 2D colour plots.

#### **INTRODUCTION**

During technological and other engineering calculation connected with thermal and hydrothermal treatment of wood materials it is necessary to have an information about thermo-physical characteristics of wood and about the impact of numerous factors on them. One of the most important such characteristic is the specific heat capacity of the wood.

The main influencing factors on the specific heat capacity are the temperature T and the wood moisture content u, including the aggregate condition of the water in the wood. The impact of these factors has been reflected in the suggested earlier by us mathematical description of the specific heat capacity of frozen and non-frozen wood (DELIISKI 1990). Our further studies have shown (DELIISKI 2003), that most precise mathematical description must include also the impact of the fiber saturation point of wood species  $u_{\rm fsp}$  and the dependence of  $u_{\rm fsp}$  from T.

Aim of the present paper is to take into account the impact of the temperature on  $u_{\rm fsp}$  in the mathematical description of the specific heat capacity of frozen wood and to compute and visualize the defrosting process in poplar logs with the help of the updated description.

# MATHEMATICAL DESCRIPTION OF THE SPECIFIC HEAT CAPACITY OF FROZEN WOOD

The ice in the wood is formed by the freezing of both the hygroscopically bounded and free water in the materials. Both the formation and the thawing of the ice as a result of the hygroscopically bounded or free water in the wood take place at different temperature ranges. The mathematical description of the specific heat capacity of frozen wood, which has been made by us, reveals the facts determined experimentally by CUDINOV (1966) in his dissertation for Dr.Sc., that the thawing of the ice from the situated in the wood free water occurs in the temperature range between 271.15 K and 272.15 K, and the thawing of the ice from the situated in the wood bounded water depends on the temperature, which is  $T \le 271.15$  K. Besides this, at T < 271.15 K, a certain portion  $u_{nfw} = f(T)$  from the bounded water is found in a non-frozen state.

Consequently during the calculation and control of the wood defrosting processes it makes sense to use the so called effective specific heat capacity of the wood  $c_{we}$ , which is equal to

$$c_{\rm we} = c_{\rm w} + c_{\rm bw} \quad @ \quad u > u_{\rm nfw} \& T \le 271,15 \text{ K},$$
 (1)

$$c_{\rm we} = c_{\rm w} + c_{fw}$$
 @  $u > u_{\rm fsp}$  & 271.15 K < T  $\leq$  272.15 K, (2)

where  $c_{we}$  is the effective specific heat capacity of the wood, J.kg<sup>-1</sup>.K<sup>-1</sup>;

 $c_{\rm w}$  - specific heat capacity of the frozen wood itself, J.kg<sup>-1</sup>.K<sup>-1</sup>;

 $c_{\rm bw}$  - specific heat capacity of the ice, which is formed by the freezing of the bounded water in the wood, J.kg<sup>-1</sup>.K<sup>-1</sup>;

 $c_{\rm fw}$  - specific heat capacity of the ice, which is formed by the freezing of the free water in the wood, J.kg<sup>-1</sup>.K<sup>-1</sup>;

u – wood moisture content, kg.kg<sup>-1</sup>;

 $u_{\rm fsp}$ - fibre saturation point of the wood, kg.kg<sup>-1</sup>;

 $u_{nfw}$ - specific amount of the non-frozen bounded water in the wood during the thawing of the ice in it, kg.kg<sup>-1</sup>; T – temperature, K.

The mathematical description of the specific heat capacity of the frozen wood  $c_w$  during defrosting of the wood has been done earlier by us (DELIISKI 1990) using the experimentally determined in the dissertations by KANTER (1955) and CUDINOV (1966) data for its change as a function of t and u. This data for  $c_w(T, u)$  finds a wide use in both the European (SHUBIN 1990, POZHGAY ET AL. 1997, TREBULA AND KLEMENT 2002, VIDELOV 2003) and the American specialized literature (STEINHAGEN 1986,

KHATTABI AND STEINHAGEN 1992) when calculating various processes of the thermal processing of wood.

In (DELIISKI 1990, 2003) the following equations for determining of  $c_w$ ,  $c_{bw}$  and  $c_{fw}$  during wood defrosting have been derived:

$$c_{\rm w} = K_{\rm w} \frac{526 + 2,95T + 0,0022T^2 + 226\,\mathrm{l}u + 1976u_{\rm nfw}}{1 + u},\tag{3}$$

$$K_{\rm w} = 1,06 + 0,04u + \frac{0,00075(T - 271,15)}{u_{\rm nfw}},\tag{4}$$

$$u_{\rm nfw} = 0.12 + (u_{\rm fsp} - 0.12) \exp[0.0567(T - 271.15)]$$
  
@  $T \le 271.15$  K, (5)

$$c_{\rm bw} = 1,893810^4 \left( u_{\rm fsp} - 0,12 \right) \frac{\exp[0,0567(T - 271,15)]}{1 + u}$$
  
@  $u > u_{\rm nfw} \& T \le 271,15 \text{ K},$  (6)

$$c_{\rm fw} = 3,34.10^5 \, \frac{u - u_{\rm fsp}}{1 + u}$$
  
@  $u > u_{\rm fsp} \& 271.15 \ {\rm K} < T \le 272.15 \ {\rm K}$ . (7)

### IMPACT OF THE FSP ON THE SPECIFIC HEAT CAPACITY OF FROZEN WOOD

Based on the results of wide experimental investigations, STAMM (1964) suggests the following equation, which reflects the influence of the temperature on the fiber saturation point of the non-frozen wood:

$$u_{\rm fsp} = u_{\rm fsp}^{293.15} - 0.001(T - 293.15), \qquad (8)$$

where  $u_{\text{fsp}}^{293.15}$  is the fibre saturation point at T = 293.15 K, i.e. at 20 °C, kg.kg<sup>-1</sup>.

Since equation (8) is generally accepted in the specialized literature, then during the mathematical description of the specific heat capacity of frozen

wood, this equation is used for the reflection of the influence of T on  $u_{fsp}$  for wood at t < 0 °C after the occurrence of complete thawing of the ice, formed from the free and bounded water in the wood, i.e:

$$u_{\rm fsp} = u_{\rm fsp}^{293.15} - 0.001(T - 293.15) \quad @ T > T_{\rm dfr} \,.$$
(9)

where  $T_{\rm dfr}$  is the temperature of complete thawing of the ice, equals to

$$T_{\rm dfr} = 271.15 + \frac{\ln \frac{u_{\rm nfw} - 0.12}{u_{\rm fsp} - 0.12}}{0.0567} \quad @ \quad 0.12 \ \rm kg.kg^{-1} \le u = u_{\rm nfw} < u_{\rm fsp}^{271.15} , \quad (10)$$

$$T_{\rm dfr} = 271.15 \quad @ \quad u \ge u_{\rm fsp}^{271.15}$$
 (11)

While the thawing of the ice from free and bounded water is taking place, a constant value of  $u_{\rm fsp}$  is used in the description of the specific heat capacity, which the wood has at the temperature of complete thawing of the ice  $T_{\rm dfr}$ , i. e.:

$$u_{\rm fsp} = u_{\rm fsp}^{293.15} - 0.001(T_{\rm dfr} - 293.15) \ @ T \le T_{\rm dfr}.$$
(12)

On Fig. 1 with an inclined line the computed according to equation (8) change in  $u_{\rm fsp}$  is shown for poplar wood, which does not contain ice depending on *t*. The computed by equation (12) horizontal lines with  $u_{\rm fsp}$  = const cross on the figures the inclined line at temperatures, which correspond to the calculated according to equation (10) and (11) values of  $t_{\rm dfr}$  for the shown in the legend on Fig. 3 values for *u* at 0.15 kg.kg<sup>-1</sup>  $\leq u \leq u_{\rm fsp}^{271.15}$ .

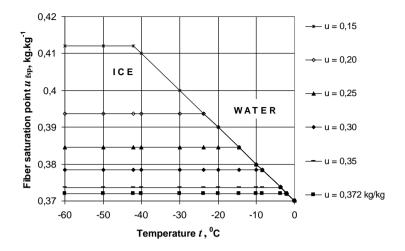


Figure 1: Change in ufsp during defrosting of poplar wood, depending on t and u

#### SOFTWARE PROGRAMM FOR COMPUTATION OF THE SPECIFIC HEAT CAPACITY OF FROZEN WOOD DURING ITS DEFROSTING

For the computation of specific heat capacity of frozen wood according to equations (1)  $\div$  (12) a software program has been prepared in FORTRAN. which has been input in the developed by Microsoft calculation environment of Visual Fortran Professional. With the help of the program computations have been made for the determination of  $c_{w}$  and  $c_{hw}$  in the ranges 213.15 K  $\leq T \leq$  271.15 K, i.e.  $-60^{\circ}C \leq t \leq -2^{\circ}C$  and  $0.15 \leq u \leq 1.2$ kg.kg<sup>-1</sup>. As an example, the specific heat capacity of often used in the production poplar (Populus alba plywood *L*.) wood with  $u_{\rm fsp}^{293.15} = 0.35 \,\text{kg.kg}^{-1}$  (VIDELOV 2003, DELIISKI 2003) has been calculated below.

On Fig. 2 and Fig. 3 the calculated change in  $c_w$  and  $c_{bw}$  of frozen poplar wood depending on *t* and *u* are presented respectively. On Fig. 4 the change in  $c_{fw}$  depending on *u* is presented.

The analysis of the graphs on Fig. 2, Fig. 3, and Fig. 4 leads to the following conclusions:

1. The increase of u at a given value of t causes a proportional increase in  $c_w$  and a decrease in  $c_{hw}$ .

2. The increase of t causes a linear increase in  $c_w$  and an exponential increase in  $c_{hw}$ .

3. The increase in *u* causes a non-linear increase in  $c_{\rm fw}$ . The value of  $c_{\rm fw}$  for the poplar wood with u = 0.6 kg.kg<sup>-1</sup> at  $t = t_{\rm dfr} = -2$  °C is greater than the values of  $c_{\rm w}$  and  $c_{\rm bw}$  correspondingly approximately 20 times and 16 times. At u = 1.2 kg.kg<sup>-1</sup> at  $t = t_{\rm dfr} = -2$  °C the value of  $c_{\rm fw}$  is greater than the values of  $c_{\rm w}$  and  $c_{\rm bw}$  correspondingly approximately 51 times and 58 times.

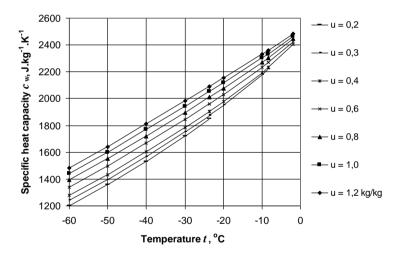


Figure 2: Change in cw of frozen poplar wood depending on t and u

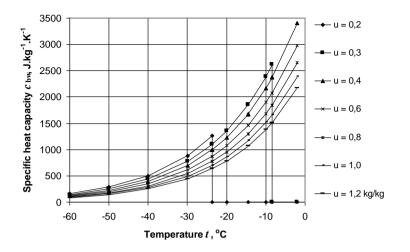


Figure 3: Change in cbw of frozen poplar wood depending on t and u

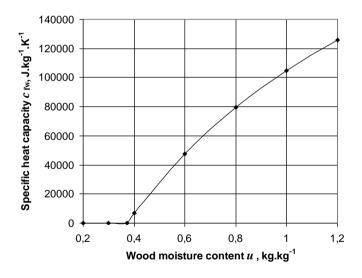


Figure 4: Change in cfw of frozen poplar wood depending on u

4. For a given value of u if the wood moisture content is within the range  $u_{nfw} < u \le u_{fsp}$ , at temperature  $t = t_{dfr}$ , at which the thawing of the hygroscopical ice finishes, a jump takes place in  $c_w$  (to  $c_w$  of the non-

frozen wood, which is not shown on Fig.2) and in  $c_{\rm bw}$  to  $c_{\rm bw} = 0$ . This jump is explained by the phase transition into water of the very last portion of the hygroscopical ice in the wood. With the increasing of u in the hygroscopic range the jump in the dependences  $c_{\rm w}(t,u)$  and  $c_{\rm bw}(t,u)$  moves to larger values of  $t = t_{\rm dfr}$  because of the fact, that at larger values of u the thawing of the ice from the bounded water ends at higher temperatures. For the poplar wood  $t_{\rm dfr} = 23.7$  °C at u = 0.2 kg.kg<sup>-1</sup> and  $t_{\rm dfr} = 8.4$  °C at u = 0.3 kg.kg<sup>-1</sup>.

### COMPUTATION AND VISUALIZATION OF 2D NON-STATIONARY TEMPERATURE DISTRIBUTION IN POPLAR LOGS

The above created mathematical description of specific heat capacity of frozen wood is introduced in the earlier suggested by us non-stationary model of defrosting processes in cylindrical wood materials (DELIISKI 2009), in which until now the influence of T on  $u_{\rm fsp}$  does not participate. The updated model has been solved out with the help of explicit schemes of the finite difference.

For solution of the updated model a software program has been prepared in the calculation environment of Visual Fortran Professional, which is a part of the office-package of Windows. With the help of the program as example computations have been made for the determination of 2D change the temperature in subjected to defrosting frozen poplar log with radius R = 0.2 m, length L = 0.8 m, initial wood temperature  $t_0 = -40$  °C and wood moisture content u = 0.6 kg.kg<sup>-1</sup> during its 16 hours of thermal

treatment in agitated hot water with  $t_{\rm m} = 80$  °C.

On Fig. 5 the computed change in the surface temperature of the logs, which is equal to  $t_{\rm m}$ , and also in the temperature in 4 characteristic points in the  $^{1}\!/_{4}$  of the longitudinal section of logs (because of its symmetry to the rest  $^{3}\!/_{4}$  of the section) containing ice both from bounded and free water is shown. The four characteristic points in the log's section have the following coordinates related to the log's surfaces: (R/2,L/4), (R/2,L/2), (R,L/4) and (R,L/2–the section's central point).

On the curves of situated on the log's axis characteristic points with coordinates (R, L/4) and (R, L/2) on Fig. 5 the specific almost horizontal

sections of retention of the temperature for a long period of time in the range from -2 °C to -1 °C can be seen, while in these points a complete thawing of the ice from the free water in the wood occurs. Such retention of the temperature has been observed in wide experimental studies during the defrosting process of pine logs containing ice from the free water (STEINHAGEN 1986, KHATTABI AND STEINHAGEN 1992).

On the Fig. 6 are shown colour plots, which illustrate the temperature distribution in <sup>1</sup>/<sub>4</sub> of the longitudinal section of the poplar log with  $t_0 = -40$  °C and u = 0.6 kg.kg<sup>-1</sup> after duration  $\tau = 4$  h and  $\tau = 8$  h of the

wood defrosting process at medium temperature  $t_{\rm m} = 80^{\circ}$  C.

On the plots of Fig. 6 it can be seen that during the defrosting of the log, which contains ice from the free water, the usual smoothness of the border between adjacent temperature zones in the legend of this figure is disturbed only in the temperature zones from -8 °C to 0 °C and from 0 °C to 8 °C. A reason for this is the shown in the analysis of Fig. 5 above retention of the temperature into the central points of the log for a too long period of time in the range from -2 °C to -1 °C, while the ice in them, formed from the freezing of the free water in the wood, is completely thawed. While the points with not completely thawed ice are still located in the colour area from -8 °C to 0 °C, their adjacent points from the calculation mesh after the complete thawing of the ice go into the zone from 0 °C to 8 °C.

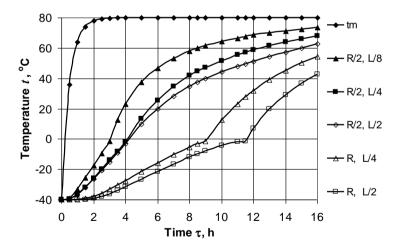
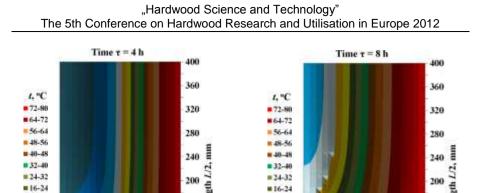


Figure. 5: Change in t in the longitudinal section of beech log with R = 0.2 m, L = 0.8 m,  $t_0 = -40 \text{ }^{\circ}C$ , and  $u = 0.6 \text{ kg.kg}^{-1}$  during its defrosting at  $t_m = 80 \text{ }^{\circ}C$ 



8-16

0.8

-8-0

-16--8

-24-16

-32--24

-40--32

200 160

120 80

Radius R. mm

160

120

80

40

0

40

160 5

120

80

40

80

Radius R. mm

40 0

120

8.16

-8-0

-16-8

-24-16

-32-24

-40-32

200 160

0.8

Figure 6: 2D color plots with temperature distribution in <sup>1</sup>/<sub>4</sub> of the longitudinal section of poplar log with  $t_0 = -40$  °C and u = 0.6 kg.kg<sup>-1</sup> after duration  $\tau = 4$  h and  $\tau = 8$  h of the wood defrosting process at  $t_m = 80$  °C

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#### CONCLUSIONS

In the present paper an approach for the computation of the effective specific heat capacity  $c_{\rm we}$  of frozen wood during its defrosting has been suggested. The approach takes into account the physics of the process of thawing of the ice, which is created in the wood by both the hygroscopically bounded and the free water. It reflects for the first time also the influence of the fiber saturation point  $u_{\rm fsp}$  of the separate wood species on the value of their  $c_{\rm we}$  during wood defrosting and the influence of the temperature on the  $u_{\rm fsp}$  of frozen wood.

The change of the specific heat capacity of frozen poplar wood itself, and the specific heat capacity of the ice, which is created in it by the freezing of the

bounded and the free water depending on wood moisture content and temperature have been calculated according to the approach.

The received results can be used in different technological and energetic calculation of the wood defrosting processes and in the systems for model based automatic control of such processes. As an example, the developed mathematical description of  $c_{\rm we}$  has been input in the suggested earlier by the author non-stationary model of defrosting processes in cylindrical wood materials. The computed with the help of this model change in the transient temperature distribution in  $\frac{1}{4}$  of the longitudinal section of subjected to defrosting poplar frozen log is graphically presented and visualized with the help of 2D colour plots.

# REFERENCES

CHUDINOV, B.S. (1966) Theoretical research of thermo physical properties and thermal treatment of wood. Dissertation for Dr.Sc., SibLTI, Krasnoyarsk, USSR.

DELIISKI, N. (1990) Mathematische Beschreibung der spezifischen Wärmekapazität des aufgetauten und gefrorenen Holzes. *Proceedings of the* 8<sup>th</sup> International Symposium ''Fundamental Research of Wood'', Warszawa, pp. 229-233.

DELIISKI, N. (2003) Modelling and technologies for steaming wood materials in autoclaves. Dissertation for Dr.Sc., University of Forestry, Sofia.

DELIISKI, N. (2009) Computation of the 2-dimensional transient temperature distribution and heat energy consumption of frozen and non-frozen logs. *Wood Research* **54**(3), 67–78.

KANTER, K. R. (1955) Investigation of the thermal properties of wood. Dissertation, MLTI, Moscow, USSR.

KHATTABI, A., STEINHAGEN, H. P. (1992) Numerical solution to twodimensional heating of logs. *Holz Roh- Werkstoff* **50**(7-8), 308-312.

POZHGAY, A. ET AL. (1997) *Structure and properties of wood*. 2<sup>nd</sup> edition, Priroda a.s., Bratislava.

SHUBIN, G. S. (1990) *Drying and thermal treatment of wood*. Publishing Company "Lesnaya promyshlennost", Moskow, URSS.

STAMM, A. J. (1964) *Wood and cellulose science*. The Ronald Press Company, New York.

STEINHAGEN, H. P. (1986) Computerized finite-difference method to calculate transient heat conduction with thawing. *Wood Fiber Science* **18**(3), 460-467.

TREBULA, P., KLEMENT, I. (2002) Drying and hydrothermal treatment of wood. TU Zvolen.

VIDELOV, H. (2003) Drying and thermal treatment of wood. University of Forestry, Sofia.

# Comparison of physical properties of heat treated and untreated hornbeam wood, beech wood, ash wood and oak wood

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**Key words:** hornbeam, beech, ash, oak, heat treatment, density, dimensional stability

#### ABSTRACT

Heat treatment is an alternative method for improving durability and dimensional stability of wood with no use of chemical additives. When we heat wood chemical changes are starting to take place inside the wood structure. Result of these changes is increased durability and dimensional stability of wood. In this thesis is shown mutual comparison between experimental results of physical properties of heat treated and untreated hornbeam wood, beech wood, ash wood and oak wood. Results of physical properties of heat treated hornbeam wood, beech wood, ash wood and oak wood found that their average value is lower and significantly different from average values of physical properties of untreated wood.

#### INTRODUCTION

Heat treatment is procedure of changing chemical structure in cell wall of wood only with influence of heath, pressure and eventually moisture without intaking any chemicals. Procedure of heat treatment is mostly conducting in reaction cylinder on temperatures between 150 °C and 260 °C without presence of oxygen (Rapp and Sailer, 2001; Yildiz *et al.* 2003; Kotilainen, 2000) or between 120 °C and 180 °C according to Patzelt *et al.* (2002). Treatment time spans between 15 minutes and 24 hours, depending on the type of the process, wood species, stock dimensions, initial moisture content, and the desired level of alteration of mechanical properties, resistance against biological deterioration, and dimensional stability of the product (Emmler and Scheiding, 2007).

Heat treatment of wood reduces its hygroscopicity, improves dimensional stability, enhances resistance against biological deterioration,

and contributes to uniform colour change from original to dark brownish tones (Stamm *et al.* 1937; Kollmann *et al.* 1975; Hill, 2006; Tjeerdsma *et al.* 1998). The disadvantages that have to be dealt with are reductions in various kinds of mechanical properties and long-term colour stability.

The objective of this research is to build knowledge of the interaction between untreated wood material properties and heat treated wood material. It is difficult to control and affect the intrinsic material properties of wood. However, it is possible to make selections and choose the most appropriate material and to reject unsuitable material. A prerequisite for this is a sufficient knowledge of relevant properties of wood. Compiled conclusions will contribute to the knowledge base on which decisions can be made that will enable the optimal choice of material and process control to achieve the desired quality in end products.

# MATERIALS AND METHODS OF RESEARCH

Research was conducted on four different species of wood: hornbeam, beech, ash and oak. One heart board, length of 2 meters was taken from each wood species. The middle of heart boards was at breast height (1,3 m). Heart boards were sawn across the middle in transverse section. Samples for determination physical properties of untreated wood were made from the part beneath breast height. The parts of heart boards above breast height were heath treated at temperature of 200 °C for 48 hours. Whole process of heating and cooling chamber lasted for 72 hours.

After finishing heath treatment, samples for determination of physical properties were made from heath treated boards. Samples were sawn from upper (breast height) part of boards down to root swelling so that the position of untreated and treated samples for determination of physical properties would be close to each other.

Heart boards of untreated wood were dried naturally too approximately 12 % of water content, and heath treated boards were 4% water content after treatment.

Testing's of physical properties: density in absolutely dry condition, radial shrinkage and tangential shrinkage were conducted by valid European standards.

The most significant physical properties that are showing the greatest difference in heat treatment process apropos those who are most important for usage of heath treated wood are given in this research. These physical properties indicate higher dimensional stability of heath treated wood in relation to untreated wood.

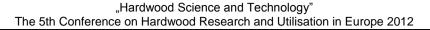
# **RESULTS AND DISCUSSION**

Mean values of density in absolutely dry condition, radial shrinkage and tangential shrinkage of untreated and heat treated hornbeam wood, beech wood, ash wood and oak wood are shown in Table 1. The rest statistical values of hornbeam wood, beech wood and ash wood are available in scientific papers from Govorčin *et al.* (2009) and Sinković *et al.* (2011). Statistical values of oak will be available in one of the upcoming articles from the same authors or if someone is particularly interested in results, original date can be obtained from authors. Results in published articles show significant difference between researched properties of untreated and heat treated wood. All values of mentioned properties of untreated wood are higher in relation to the heat treated wood (Govorčin *et al.* 2009 and Sinković *et al.* 2011).

 Table 1 Survey of average values for density in absolutely dry condition, radial shrinkage and tangential shrinkage of untreated and heat treated wood.

U	ntreated woo	bd		Heat treated wood		
$\rho_{o}$	$\beta_{r max}$	$\beta_{t max}$	wood species	$\beta_{t max}$	$\beta_{r max}$	ρ <sub>o</sub>
g/cm <sup>3</sup>	%	%		%	%	g/cm <sup>3</sup>
0,716	8,02	9,86	hornbeam	5,27	3,65	0,666
0,680	5,78	12,60	beech	10,23	5,38	0,627
0,655	6,96	9,00	ash	3,45	2,15	0,600
0,681	5,34	5,90	oak	4,79	2,78	0,672

Key:  $\rho_o$  - density in absolutely dry condition,  $\beta_{r\ max}$  - radial shrinkage,  $\beta_t\ _{max}$  - tangential shrinkage



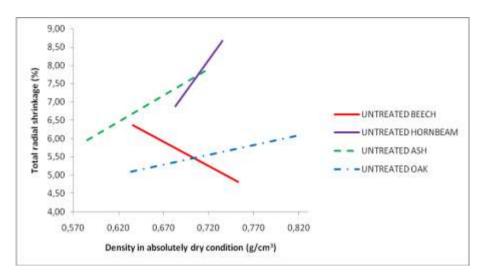


Figure 1 Relation between density in absolutely dry condition and total radial shrinkage for untreated beech wood, hornbeam wood, ash wood and oak wood.

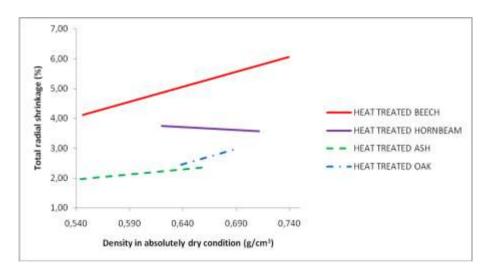


Figure 2 Relation between density in absolutely dry condition and total radial shrinkage for heat treated beech wood, hornbeam wood, ash wood and oak wood.

According to Figures 1 and 2 beech wood has changed trend of relation between density in absolutely dry condition and total radial shrinkage between untreated and heat treated wood.

Untreated beech wood has decreasing trend of relation between density in absolutely dry condition and total radial shrinkage, and heath treated beech wood has growth trend.

For untreated hornbeam wood by increasing density in absolutely dry condition total radial shrinkage increases, and for heat treated hornbeam wood total radial shrinkage decreases. For untreated and heath treated ash wood and oak wood by increasing density in absolutely dry condition total radial shrinkage increases.

Heat treated hornbeam wood has an average value of total radial shrinkage by 120% smaller compared to the untreated hornbeam wood. Total radial shrinkage of heat treated beech wood is by 7% smaller compared to the untreated beech wood and total radial shrinkage of heat treated oak wood is by 92,1% smaller compared to the untreated beech wood.

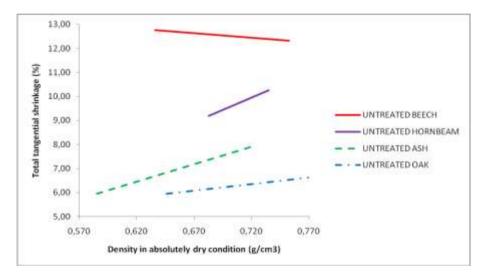


Figure 3 Relation between density in absolutely dry condition and total tangential shrinkage for untreated beech wood, hornbeam wood, ash wood and oak wood.

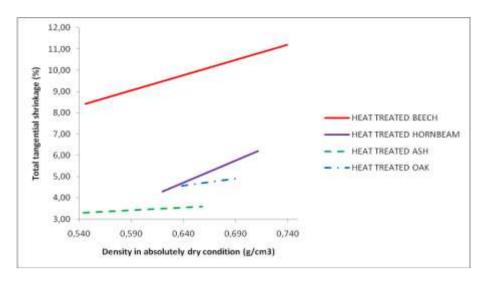


Figure 4 Relation between density in absolutely dry condition and total tangential shrinkage for heat treated beech wood, hornbeam wood, ash wood and oak wood.

Untreated beech wood has decreasing trend of relation between density in absolutely dry condition and total tangential shrinkage, and heath treated beech wood has growth trend. Both untreated and heat treated hornbeam wood, ash wood and oak wood have growth trend of relation between density in absolutely dry condition and total tangential shrinkage.

Heat treated hornbeam wood has an average value of total tangential shrinkage by 87,1% smaller compared to the untreated hornbeam wood. Total radial shrinkage of heat treated beech wood is by 23,2% smaller compared to the untreated beech wood and total radial shrinkage of heat treated oak wood is by 23,2% smaller compared to the untreated beech wood.

#### CONCLUSIONS

Heath treated hornbeam wood, beech wood, ash wood and oak wood have decreased density in absolutely dry condition, radial and tangential shrinkage in relation to the same physical properties of mentioned untreated wood species.

There is a change in trend of relation between density in absolutely dry condition and total radial shrinkage and total tangential shrinkage for heat treated beech. Untreated beech has decreasing trend of total radial and total tangential shrinkage in relation to increase in density in absolutely dry condition. Heat treated beech has growth trend of total tangential and total radial shrinkage in relation to increase in density in absolutely dry condition.

According to researched results of radial and tangential shrinkage of hornbeam wood, beech wood, ash wood, and oak wood, heath treated wood has greater dimensional stability in relation to untreated wood.

# REFERENCES

- 1. Emmler, R.; Scheiding, W. 2007: Darker shades of wood: Thermally modified timber (TMT) as a new material for parquet floorings. European Coatings Journal April: 106-111.
- Govorčin, S.; Sinković, T.; Hrčka, R. 2009: Usporedba tehničkih svojstava termički modificiranog i recentnog drva jasena (*Fraxinus excelsior* L.). Šumarski list CXXXIII (2009)(3-4)185-191.
- 3. Hill, C. 2006: Wood Modification: Chemical, Thermal and Other Processes. John Wiley and Sons, Ltd, 99-127.
- Kollmann, F.P.; Kuenzi, E.W.; Stamm, A.J. 1975: Principles of Wood Science and Technology – Wood Based Materials. Springer – Verlag, New York, Heidelberg, Berlin, 118-120.
- 5. Kotilainen, R., 2000: Chemical Changes in wood during heating at 150-260 °C. Ph.D. Thesis, Javaskyla University, Finland.
- Patzelt, M.; Stigl, R.; Teischinger, A. 2002: Thermische Modifikation von Holz und deren Einfluss auf ausgewählte Holzeigenschaften. Modifiziertes Holz: Eigenschaften und Markte, Lignovisionen Band 3, 2002, 101-147.
- 7. Rapp, A.O.; Sailer, M. 2001: Oil heath treatment of wood, Drvna industrija, 52:2, 63-70.
- Sinković, T.; Govorčin, S.; Sedlar, T. 2011: Usporedba fizikalnih svojstava neobrađene i toplinski obrađene bukovine i grabovine. Drvna industrija, 62(4)283-290.

- Stamm, A.J.; Hansen, L.A. 1937: Minimizing wood shrinkage and swelling. Effect of Heating in Various Gases. Industrial & Engineering Chemistry, July 1937.
- Tjeerdsma, B.F.; Boonstra, M.; Pizzi, A.; Tekely, P.; Militz, H., 1998: Caracterisation of termally modifi ed wood: molecular reasons for wood performance improvement. Holz Roh-Werkst 56 (3): 149-153.
- 11. Yildiz, S.; Yildiz, Ü.; Çolakođlu, G.; Derya, E.; Temýz Ali, G. 2003: The effects of heat treatment on the specific gravity of beech and spruce wood, IRG 34th Annual Meeting in Brisbane, Australia, IRG/WP 03-40254.

# Properties of trunk and briarwood of tree heath (*Erica arborea* L.) from island Rab

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**Key words:** Tree heath (*Erica arborea* L.), briarwood, trunk, physical properties, mechanical properties

#### ABSTRACT

Tree heath (*Erica arborea* L.) is an evergreen shrub that grows mainly in Mediterranean region. This species tends to grow in areas such as macchia shrub lands, dry evergreen scrublands, forest roadsides and forest outskirts which have a lot of light and sun though daytime. Tree heath is not a commercial timber species, it occurs as a result of forest roads and forest fireroads construction. This wood species is interesting because of its briarwood. Briarwood is tumour like outgrow that develops between root and trunk and it's commonly used in making bowls of tobacco smoking pipes and knife handles. The trunk can also be used for variety of products because of its relatively good mechanical properties and nice colour and texture. Material for this study was taken from tree heath (*Erica arborea* L.) shrubs growing on island of Rab in Croatia. In this study density and dimensional stability of briarwood and trunk of tree heath were investigated. Also some mechanical properties of trunk such as bending strength and compression strength parallel to the grain were studied.

#### AIM OF RESEARCH

Knowing technological characteristics of wood is important postulate for rational usage of wood recourses. It is important to define anatomical, chemical, physical and mechanical properties of some wood species to determine its technological characteristics.

The aim of this research was to define some physical and mechanical properties of tree heath (*Erica arborea* L.) for further and more complete determination of its technological characteristics. Two different

parts of tree heath were studied. One was the trunk and the other was its root also known as briarwood. Briarwood was studied for its physical properties; density in absolutely dry condition, nominal density, and total volume shrinkage. Only total volume shrinkage was studied because briarwood has specific and complex structure and it is impossible to determent direction of grain on specimens. Trunk was studied for some of its physical and mechanical properties. The following physical properties of trunk were studied: density in absolutely dry condition, nominal density, total radial, tangential and volume shrinkage. Mechanical properties that were studied are ultimate compression strength parallel to the grain and ultimate bending strength.

#### MATERIAL AND METHODS OF RESEARCH

The tree heath (*Erica arborea* L.) is a shrub or small evergreen tree (Hirc, 1891) that grows mainly in Mediterranean region. These are particularly difficult conditions for most plants to grow in as the soil in the mountainous clime is often dry and rocky, but the hearty briar roots can work their way through the tiniest crevices or slowly break apart rock to reach the rare soil. It has a typical height of 1 to 4meters, with some specimens reaching even 7 meters. This species tends to grow in areas such as macchia shrub lands, dry evergreen scrublands, forest roadsides and forest outskirts which have a lot of light and sun though daytime. It occurs as a result of forest roads and forest fire roads construction. The tree heath grows bright green needle-like leaves and each spring its greyish-white flowers bloom (Marčić, 1918), producing a delightful honey scent. Between the acidic and rocky soil it grows in and the long summer droughts it endures, this small trees root develops outgrow that is called briarwood.

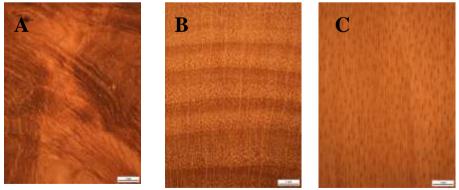


Figure 1 Macroscopic preview of briarwood and trunk wood: A – Briarwood, B – Trunk wood cross section, C – Trunk wood tangential section

Briarwood (Tsoumis, 1988) is tumor like outgrow that develops between root and trunk and its commonly used in making bowls of tobacco smoking pipes, knife handles and it was used by blacksmiths for large wood heating power (Lasman, 1906). The use of briar as an ideal material to make bowls of pipes dates back to at least the 1850s. The bush must be at least 50 years old for the briar to grow to the necessary size, about the size of a football, weighing around 3 kg. Briarwood is a material that never alters. It is extremely hard and heat-resistant and in addition has a lovely grain that can give a sense of flames and never repeats itself. Each piece is unique.

The trunk of tree heath can also be used for variety of products because of its relatively good mechanical properties and interesting esthetic properties such as nice colour and texture.

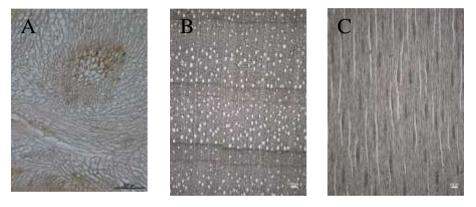


Figure 2 Photomicrographs of briarwood and trunk wood: A - Briarwood (20×), B - Trunk wood cross section (4×) and C - Trunk wood tangential section (4×)

On Figure 1 macroscopic preview of briarwood and trunk is presented. Figure 2 presents photomicrographs of briarwood and trunk wood. On both figures the difference in normal structure of trunk wood in comparison to the irregular structure of briarwood is shown.

Material for this study was taken from three tree heath (*Erica arborea* L.) shrubs growing on island of Rab in Croatia. These shrubs were 20 to 30 years old and (horizontal) diameter of the tumors was about 30 to 50 cm. Total height of shrubs was about 5 to 6 meters. The shrubs were sampled for trunk and tumor as it's shown in Figure 3 and specimens were prepared to study test or examine the physical and mechanical properties.



Figure 3 Briarwood and trunk wood (Erica arborea L.)

Physical properties of trunk and tumor were determined on sharpedged samples dimensions  $20 \times 20 \times 25$  mm (R×T×L). Samples were then soaked in water during the period in which they exceeded water content higher then fiber saturation point. After attaining wanted water content, the samples were dried on temperature of  $103\pm2^{\circ}$ C until they reached constant mass. As well, after attaining absolutely dry condition measurements were again completed and data were processed, all according to valid norms.

Ultimate compression strength parallel to the grain was determined on sharp-edged samples of trunk with dimensions  $20 \times 20 \times 40$  mm (R×T×L) according to the standard HRN ISO 3787:1999, and ultimate bending strength on samples with dimensions  $20 \times 20 \times 300$  mm (R×T×L) according to the standard HRN ISO 3133:1999.

#### **RESULTS OF RESEARCH**

#### **Physical properties**

Table 1 Statistical values of density in absolutely dry condition, nominal density and total	,
radial, tangential and volume shrinkage of tree heath trunk wood and briarwood.	

Tree heath (trunk wood)			Tree heath (briarwood)					
ρ <sub>0</sub>	ρ <sub>y</sub>	β <sub>rmax</sub>	$\beta_{tmax}$	$\beta_{vmax}$		$\beta_{vmax}$	ρ <sub>y</sub>	ρ <sub>0</sub>
g/cm <sup>3</sup>	g/cm <sup>3</sup>	%	%	%		%	g/cm <sup>3</sup>	g/cm <sup>3</sup>
21	21	21	21	21	N	137	137	136
0,743	0,627	4,4	6,0	12,4	MIN	10,4	0,573	0,698
0,770	0,648	6,2	10,0	15,9	AVE	20,5	0,666	0,833
0,803	0,673	9,9	13,1	17,8	MAX	92,4	0,732	0,973
0,0191	0,0149	1,6109	1,9939	1,2384	SD	7,2126	0,0370	0,0482
0,0004	0,0002	2,595	3,975	1,534	VAR	52,021	0,0014	0,0023

Key:  $\rho_o$  - density in absolutely dry condition,  $\rho_y$  – nominal density,  $\beta_{r\ max}$  - total radial shrinkage,  $\beta_{t\ max}$  - total tangential shrinkage and  $\beta_{v\ max}$  - total volume shrinkage

According to Table 1 mean values of researched physical properties of trunk wood are smaller in all segments then the ones of briarwood. Mean value of density in absolutely dry condition of briarwood is higher by 7,6 % then the same value of density in absolutely dry condition of trunk wood. Mean value of nominal density of briarwood is only by 2,7 % higher than the same value of trunk wood. Mean values of total volumetric shrinkage of briarwood is higher by 22,4 % then mean value of trunk wood.

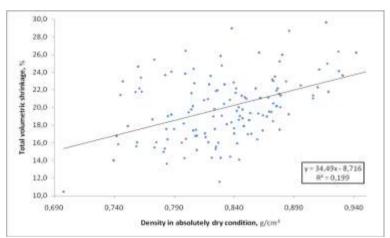


Figure 4 Relation between total volumetric shrinkage and density in absolutely dry condition of briarwood

Figure 4 shows growth trend of total volumetric shrinkage in relation to the density in absolutely dry condition of briarwood. The same growth trend occurs at trunk wood (Figure 5) only with smaller correlation coefficient.

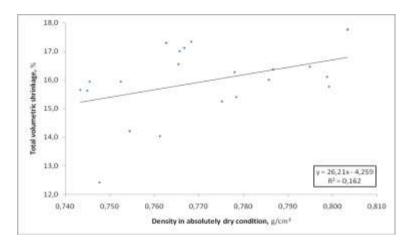


Figure 5 Relation between total volumetric shrinkage and density in absolutely dry condition of trunk wood

#### Mechanical properties

Table 2 Statistical values of density at 12% water content, ultimate bending strength at 12% water content, ultimate compression strength parallel to the grain at 12% water content of tree heath trunk wood.

	Tree heath (trunk wood)				
$\sigma_{b12\%}$	ρ <sub>12%</sub>		ρ <sub>12%</sub>	$\sigma_{c12\%}$	
MPa	g/cm <sup>3</sup>		g/cm <sup>3</sup>	MPa	
12	12	Ν	7	7	
18,8	0,863	MIN	0,864	54,9	
49,1	0,920	AVE	0,951	58,9	
110,2	1,004	MAX	1,002	61,8	
29,304	0,0405	SD	0,0525	2,910	
167,972	0,0010	VAR	0,0028	8,466	

Key:  $\rho_{12\%}$  - density at 12% water content,  $\sigma_{b12\%}$  – ultimate bending strength at 12% water content,  $\sigma_{b12\%}$  – ultimate compression strength parallel to the grain at 12% water content

According to Table 2 mean value of ultimate bending strength at 12% water content of trunk wood is 49,1 MPa. Mean value of ultimate

compression strength parallel to the grain at 12% water content of trunk wood is 58,9 MPa.

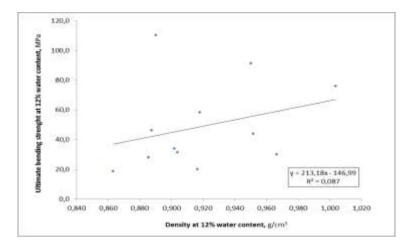


Figure 6 Relation between ultimate bending strength at 12% water content and density at 12% water content of trunk wood

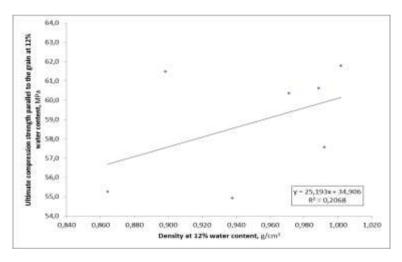


Figure 7 Relation between ultimate compression strength parallel to the grain at 12% water content and density at 12% water content of trunk wood

Figure 6 shows that ultimate bending strength at 12% water content has a growth trend with the increase in density at 12% water content of trunk wood. Figure 7 shows that ultimate compression strength parallel to the

grain at 12% water content also has growth trend with the increase in density at 12% water content.

#### CONCLUSIONS

Conducted study, measuring of samples and analysis of data that have been made on trunk of tree heath (*Erica arborea* L.) resulted in mean value of density in absolutely dry condition is  $0,770 \text{ g/cm}^3$ , and mean value of nominal density is  $0,648 \text{ g/cm}^3$ . Mean value of total radial shrinkage is 6,2%, total tangential shrinkage is 10,0 %, and mean value of total volume shrinkage is 15,9 %. Mean value of ultimate compression strength parallel to the grain at 12% water content is 58,9 MPa and mean value of ultimate bending strength at 12% water content is 49,1 MPa. Mean value of density in absolutely dry condition of briarwood is  $0,833 \text{ g/cm}^3$ , and mean value of nominal density is  $0,666 \text{ g/cm}^3$ . Studied briarwood has mean value of total volume shrinkage of 15,9 %.

#### REFERENCES

- 1. Hirc, D., 1891: Pogled u floru hrvatskoga Primorja s osobitim obzirom na šumsko drveće i grmlje. Šumarski list 15 (3) 107-118.
- 2. HRN ISO 3787:1999 Određivanje čvrstoće na tlak paralelno s vlakancima.
- 3. HRN ISO 3133:1999 Određivanje statičke čvrstoće na savijanje.
- Lasman, D., 1906: Šumarenje na otoku Rabu. Šumarski list 30 (4) 138-163.
- 5. Marčić, M., 1918: Sredozemna flora u drvoredima i parkovima na istočnim obalama Jadrana. Šumarski list 62 (5) 129-137.
- Tsoumis, G., Kezos, N., Fanariotou, I., Voulgaridis, E. and Passialis, C., 1988: Characteristics of Briarwood. Holzforschung 42 (1988) 71-77.

# Anatomical, Mechanical and Physical Properties of Two indigenous Hardwood species grown in Sudan

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**Keywords:** Anatomical, Mechanical and physical properties, *Acacia seyal var. seyal, Balanites aegyptiaca.* 

### ABSTRACT

Despite the richness of Sudan in a great diversity of tree species, the utilization of wood resources has traditionally concentrated to a few numbers of them. Most of the indigenous hardwood tree species in Sudan are used as charcoal, firewood and fuel wood due to the lake information about their properties. There is an urgent need to study the wood properties of the local raw material in order to suggest better uses for their wood than conversion to firewood and charcoal. This would not only reduce imports, but they would also provide an economic incentive to the forestry and industrial sectors of Sudan.

The present study was carried out to provide basic informations about the anatomical, mechanical and physical properties of two indigenous hardwood species grown in Sudan namely, *Acacia seyal var. seyal* and *Balanities aegyptiaca*. Both species are widely distributed and easily to grow on large areas in Sudan; their uses concentrate in charcoal, firewood and fuel wood. The wood materials were collected from different zones in Sudan. Some anatomical, mechanical as well as physical properties were investigated. In anatomical investigations, fiber length, diameter, lumen diameter as well as double wall thickness were measured from which the Runkel ratio, slenderness ratio and flexibility coefficient were obtained. Hardness Strength was measured as mechanical property. Concerning physical investigations, wood basic density was determined.

The results revealed that the wood anatomical, mechanical and physical properties of the study species may qualify them for advanced industries like

pulp and paper, fiber board and flooring industries. The results of this study could enhance the establishment of forest industry in Sudan.

## INTRODUCTION

Sudan is a country rich in forest including a large number of tree species; it encompasses more than 3156 species belonging to 1137 genera and 170 families (BROUN AND MASSAY 1929, ANDREWS, 1950, 1952, AND 1956, EL AMIN 1990). Nevertheless the utilization of wood resources has traditionally concentrated to a few numbers of species. Most of the indigenous hardwood tree species in Sudan were used as charcoal, firewood and fuel wood due to the lake information about their properties. Good examples for those species are *Acacia seyal var. seyal* Del. and *Balanities aegyptiaca* (L.) Delile. Those two species are widely distributed and easily grow on large areas in Sudan.

Wood density is the most significant property in determining wood end. Wood density is strongly effect on the yield, strength, and general quality of most of the products produced from wood. Wood anatomy is described as fundamental basis of timber utilization. A wood anatomist can suggest uses, especially for woods that are not in a commercial demand. Previous studies have shown that fiber morphology is an important indicator for end-use. For instance it affects papermaking properties of wood species. Some researchers like SAIKIA ET AL. (1997), OGBONNAYA ET AL. (1997) and VERVERIS ET AL. (2004) have successfully used the fiber derived values to assess the suitability of various fiber raw materials for pulp and paper manufacture. According to BOWYER ET AL. (2004), mechanical properties are usually the most important characteristics of wood product to be used in structural applications such as flooring and rafters, structural panel roof, wall sheathing, etc...

Wood properties studies have a special significance in countries like Sudan where only a few timbers are well known. After the oil extract in Sudan, great amounts of wood will be available for uses other than energy because oil will gradually replace wood, the main energy resource.

This research is an attempt to provide basic information on some anatomical, mechanical and physical properties of *Acacia seyal var. seyal*. and *Balanities aegyptiaca* in order to suggest best uses for them.

# MATERIALS AND METHODS

## Materials

The wood raw materials were collected randomly from 11 natural forests located in four states in Sudan in order to have good presentation for the country. According to the mean annual rainfall for ten years (2000 - 2009), the study areas were divided into two zones; zone one: with a relatively low rainfall (273mm annually, mean average rainfall), and zone two: with relatively high rainfall (the mean average rainfall is 701 mm annually).

The location and characterization of the study areas are summarised in Fig. 1, while sampling procedure is presented in Fig. 2.

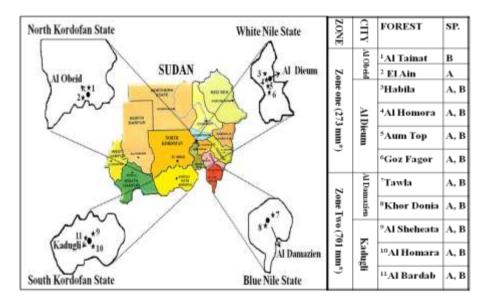


Figure 1: Location and characterization of the study areas (\*= Zone's mean annual rainfall of 10 years (2000-2009); Sp. = species; A = Acacia seyal var. seyal and B = Balanites aegyptiaca)

Three healthy and straight trees were selected randomly and cut down from each forest for each species give a total of 60 trees for both species. The tree total height, merchantable height and diameter at breast height (DBH) were measured for each tree.

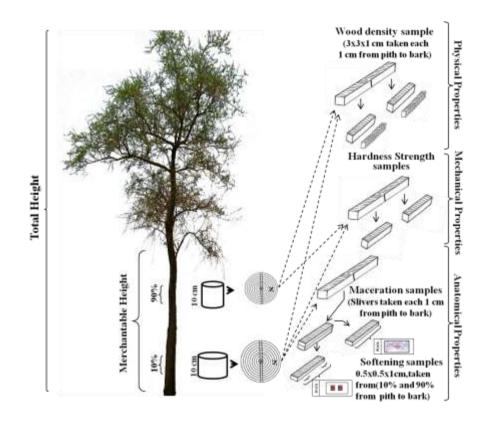


Figure 2: Sampling procedure

Two discs of 10 cm thick were obtained from each tree at 10% and 90% of the merchantable height. Afterwards the discs were cut into small samples or strips (include tree's pith) with 3 x 3 x tree diameter in cm. Two free of defect strips were taken from each 90% disc and three from 10% disc given a total of 5 strips for each tree. One strip of 90% disc and one of 10% disc for each tree were wrapped in plastic bags and stored immediately in refrigerator to keep it wet to be used in density investigation. All others strips were dried, and then the second strip of the 90% disc and one of 10% discs were used in hardness strength investigation. The third strip of 10% disc was used for the anatomical investigations.

# Methods

### Wood density

The wood basic density was measured as oven-dry mass/green volume. One wet sample from each stem height (10% and 90%) were taken and were cut from the pith (center) into two parts and each part has been sawn into small specimens with  $1x \ 3x \ 3$  cm.

The specimens green volume was determined by water replacement method, then were immediately transferred into an oven and dried until approximately the constant mass was attained. The dry weight was measured using sensitive balance. The basic density was calculated using the following formula:

$$D = \frac{W_{\text{ovendry}}}{V_{\text{green}}} \tag{1}$$

Where, D is the basic density in  $g/cm^3$ .  $W_{oven dry}$  is the dry weight in g.  $V_{green}$  is the green volume in  $cm^3$ .

## Fiber dimensions and their derived values:

Two anatomical tests were conducted, maceration test to measure fiber length, and the softening test to measure fiber diameter and lumen diameter. One air dry sample or strip from stem height 10% was selected from each tree of the study species and was separated from the pith localization into two samples (radiuses). One radius was taken to perform the anatomical properties investigations. This selected radius was then separated into two samples in middle length. The upper part was used for the maceration test by setting sampling points at centimeter intervals from pith to bark and cutting small slivers from each sampling point. The lower part was used for softening test by setting two sampling points one at 10% and the second at 90% distance from pith to bark.

The maceration procedure developed by Shultze as cited in JANE (1970) was adopted to macerate the woody materials in order to measure the fiber length. Small slivers of wood were placed in test-tubes, to which 65% nitric acid with a few crystals of potassium chlorate (KCLO<sub>3</sub>) was added and then warmed up in a water bath for about 5-10 minutes. The macerated material was washed several times by distilled water and stained by few drops of safranin dye for five minutes. After staining, the macerated material was rewashed several times with distilled water and then transferred to slides

surface. Few drops of Kaiser's glycerol gelatine were added to each slide and then covered gently with a cover slip and left to dry gradually for a day. A number of 40 fibers length were measured randomly from each sample using light microscope (model: Variant Jenamed) with an 10x ocular lens provided with a measuring scale graduated into ten equal segments and each segment is graduated into ten sub-segments.

Concerning the softening test, One sample of  $0.5 \ge 0.5 \ge 1$  cm was cut from each sampling point (10% and 90% distance from pith to bark) to prepare cross section in order to measure fibers diameter, lumen diameter and double wall thickness.

Balanites aegyptiaca's samples were softened by boiling in water for about 8-10 hours. Due to the high wood density of Acacia seyal var.seyal, softening in autoclave device method as described in (GROSSER 1971) for Bamboo species was adopted by cooking in autoclave device for 3 hours using a temperature of 140 °C and a pressure of 4-5 bars. The cooked Acacia seyal's samples were then immersed in 75% ethanol (JAGIELLA AND KÜRSCHNER1987) for a week. transverse sections 10-15µm in thickness were cut using GSL1 microtome (invented by H. Gaertner, F.H. Schweingruber & S. Luccinetti) The prepared sections from both species were dehydrated in a graded ethanol series (25%, 50% and absolute, respectively), stained with safranine dye and dehydrated again in the same graded ethanol series. The sections were then transferred carefully into slides to which few drops of glycerin gelatin were added, and then covered gently with cover slips. The slides were left 24 hours to dry.

Nikon coolpix 990 Camera fixed in light microscope (model: Variant Jenamed) which in turn was connected with PC were used to take photos from the prepared slides. The image j software was used to measure the fibers's dimension from the photos. A number of 40 fibers were selected randomly to measure fiber diameter and lumen diameter. Fibers wall thickness was calculated using the following equation:

$$WT = \frac{D - LD}{2} \tag{2}$$

Where: DWT is double wall thickness; D is diameter and LD is lumen diameter

Three derived values were also calculated using fiber dimensions: Slenderness ratio as fiber length/fiber diameter, Flexibility coefficient as (fiber lumen diameter/ fiber diameter)  $\times$  100 and

Runkel ratio as  $(2 \times \text{fiber cell wall thickness})/\text{lumen diameter}$ .

#### Hardness strength

Brinell hardness test was conducted on the basic of DIN EN 1534 to measure the hardness strength of the studied species. One conditioned at 12% sample (stripe) from each stem height (10% and 90%) were selected. Each stripe was split from the pith (center) into two radius. In order to obtain soft surface, the four sides of each radius were sanded using sanding machine. The TIRA test 28100 machine provided by a hardened steel ball with a diameter of  $10 \pm 0.01$  mm was used to perform the hardness test in the transverse and radial sections.

The following equation has been used to get the hardness:

$$H = \frac{2F}{g.\pi.D(D - (D^2 - d^2)^{1/2})}$$
(3)

Where:

Н	is the hardness,
g	is the acceleration due to gravity, in meters per second squared,
π	is the factor ", $pi'' \approx 3.14$ ),
F	is the nominal force in Newton,
D	the ball diameter in millimetres,
4	is the discussion of the improvement in million stress

d is the diameter of the impression point in millimetres.

### **RESULTS AND DISCUSSION**

Table no 1 show the mean values for the investigated wood properties. The average basic density of *Acacia seyal* and *Balanites aegyptiaca* (735 and 657, respectively) was in the range of tropical hardwoods of 400-900 kg/m<sup>3</sup> (Tissot 1985). They could be graded as medium-heavy (Bin 1970). *Acaica seyal var. seyal* density estimated in the current study was higher than those estimated by other authors like those of KHRISTOVA *ET AL*. (2004) and KHRISTOVA *ET AL*.(1998) (669-692kg/m<sup>3</sup> and 649 kg/m<sup>3</sup>, respectively). The reason may due to the yang trees used in their studies. KHRISTOVA *EL AL*. (1997) found the wood basic density of *Balanites aegyptiaca* to

be 619 kg/m<sup>3</sup> which is more or less comparable with those obtained in the current study (657 kg/m<sup>3</sup>).

Acacia seyal var. seyal density (734.95 kg/m<sup>3</sup>) is slightly above the range for commercial temperate pulpwood of 350-650 kg/m<sup>3</sup>, and that of *Balanities aegyptiaca* (657.41 kg/m<sup>3</sup>) is almost within the range.

	Stu	udy Species		
Property	Acacia seyal	Balanites aegyptiaca		
Basic density	734.95	657.41		
[kg/m <sup>3</sup> ]				
Fiber dimension				
Length [mm]	1.27	1.16		
Diameter [ µm]	10.26	13.09		
Lumen Diameter	3.70	5.44		
[µm]				
Wall Thickness	6.56	7.64		
[ µm]				
Fiber derived values				
Flexibility	35.65	41.24		
Coefficient (%)				
Runkel Ratio	1.98	1.55		
Slenderness ratio	124.04	88.74		
Hardness strength [ N	/mm <sup>2</sup> ]			
In Cross surface	84.14	86.37		
In radial surface	51.62	45.22		

 Table 1: Wood density, fiber characteristics and hardness strength mean values of the study species

The fiber lengths of both species are in the range of hardwood of 0.7-2.0 mm (ILVESSALO- PFAFFLI 1995) and considered as short according to WAGENFÜHR (1984), and Medium according to IAWA (1989) classifications. They are also within the normal range of hardwood for commercial pulping. Many authors confirmed the suitability of species with equal and event shorter fiber length than the studied species for pulp and paper making. Good examples are the species studied by KHRISTOVA EL AL. (1997), KHRISTOVA EL AL. (1998), KHRISTOVA AND KARAR (1999), KHRISTOVA EL AL. (2004) and that of DUTT AND TYAGI (2011).

These fibers were of good slenderness ratios (124.04 for *Acacia seyal* and 88.74 for *Balanites aegyptiaca*), much more than the acceptable value for papermaking of > 33 (XU ET AL. 2006). It is much bigger than almost all those of hardwood using in papermaking and also bigger than some soft wood species like *Pinus kesiya* (56.51) as sited in DUTT AND TYAGI (2011). This enhances their suitability for pulp and paper making.

The Runkel ratio of *Balanites aegyptiaca* estimated in the current study was 1.55 which is at the upper end of the acceptable range of papermaking (0.25-1.5) as sited by VALKOMER (1969), while that of *Acacia seyal var.seyal* (1.98) was out of the range. KHRISTOVA EL AL. (1997) and KHRISTOVA EL AL. (1998) confirmed the suitability of species with higher Runkel ratio for papermaking (2.9-2.5 respectively).

Nevertheless, the flexibility coefficient of both species are much lower than the acceptable value for papermaking of preferably >60 (PETRI 1952, OKEREKE 1962 AND RYDHOLM 1965). But they are comparable to those of the species studied by other authors (KHRISTOVA *EL AL*. 1997, KHRISTOVA EL AL. 1998 AND KHRISTOVA AND KARAR 1999).

The estimated hardness strength (in cross and tangential surfaces) of both species is bigger than those of *Robinia pseudoacacia* which commercially used in flooring industry. GÖHRE (1952) and KOLLMAN (1951) found the *Robinia pseudoacacia* hardness strength in cross surface to be 78.2 N/mm2 and 74 N/mm2, respectively. While those of tangential surface is estimated by 33.2 N/mm2 (GÖHRE 1952). Those values obtained from literature are smaller than the estimated values for *Acacia seyal* and *Balanites aegyptiaca* in the current study of 84.14 and 51.62 N/mm2 and 86.37 and 45.22 N/mm2 for cross and tangential surface, respectively. Also *Acacia seyal* density estimated in current study (734.95 kg/m<sup>3</sup>) is comparable with those of *Robinia pseudoacacia* of 770 kg/m<sup>3</sup> as estimated by GÖHRE (1952) and KOLLMAN (1951). While that of *Balanites* (657.41 kg/m<sup>3</sup>) is lower.

# CONCLUSION

Wood density, fiber dimensions and hardness strength of the studied species are in the normal range for hardwoods. In general wood density and fiber characteristics of the study species are compatible for paper and fiber board industries. Also their hardness strength as well as density are compatible for flooring industry.

From the results, it is obvious that the studies species can be put to better use than conversion to firewood and charcoal. Their suitability for such advanced industry would not only reduce imports, but also would provide an economic incentive to the forestry and industrial sectors of Sudan.

#### REFERENCES

ANDREWS, F. W. (1950, 1952, 1956). The Flowering Plants of the (Anglo-Egyptian) Sudan. Vol. 1, 2, and 3. The Burncle of Co., Ltd, Arbroath.

BROUN, A. F. and R. E. MASSEY (1929). Flora of the Sudan. Wellington House, London.

DUTT, D., and TYAGI, C.H. (2011). Comparison of various eucalyptus species for their morphological, chemical, pulp and paper making characteristics. Indian Journal of Chemical Technology. 18, 145-151.

EL AMIN, H.M. (1990). Trees and Shrubs of the Sudan. Ithaca Press, Exeter.

GÖHRE, K. (1952). Die Robinie und ihr Holz. Deutscher Bauernverlag, Berlin.

GROSSER, D. (1971): Beitrag zur Histologie und Klassifikation asiatischer Bambusarten. In: Mitteilungen der Bundesforschungsanstalt für Forst und Holzwirtschaft REINBECK, Nr. 85. WiedebuschVerlag, Hamburg. 321 S.

IAWA COMMITTEE (1989). IAWA list of microscopic features for hardwood identification. IAWA Bull. n.s 10 (3): 221-332.

ILVESSALO- PFAFFLI, M-S. (1995). Fiber Atlas: Identification of papermaking fibers. Springer-Verlag, Berlin.

BOWYER, J, L., SHMULSKY, R. AND HAYGREEN, J. G. (2004). *Forest Products and Wood Science – An Introduction*, pages 160–193; 228–241. Blackwell Publishing Company, fourth edition.

JAGIELLA, C., KÜRSCHNER, H., (1987). Atlas der Hölzer Saudi Arabiens. Die Holzanatomie der wichtigsten Bäume und Sträucher mit einem holzanatomischen Bestimmungsschlüssel. Dr. Ludwig Reichert Verlag, Wiesbaden. 176 S.

JANE, F.W. (1970). The Structure of Wood. 2<sup>nd</sup> ed. Completely revised by K. Wilson and D.J.B White. London.

KHRISTOVA, P., GABBIR, S., BENTCHEVA, S., AND DAFALLA, S. (1997). Soda-AQ pulping of three Sudanese hardwoods, *Tropical Sci. 37*, 176-182.

KHRISTOVA, P., GABIR, S., BENTCHEVA, S., KHIDER, T. (1998). Soda-AQ pulping of four Acacias from Sudan. Cellulose Chemistry and technology 32: 299-307.

KHRISTOVA, P., KORDSACHIA, O., AND DAFALLA, S. (2004). Alkaline pulping of *Acacia seyal*. Tropical Sci.44, 207-215.

KOLLMANN, F. (1951). Technologie des Holzes und der Holzwerkstoffe, 1.Band, Anatomie und Pathologie, Chemie, Physik, Elastizität und Festigkeit. 2.Auflage. Springer-Verlag, Berlin, Göttingen, Heidelberg.

OGBONNAYA, C.I., ROY-MACAULEY, H., NWALOZIE, M.C., ANNEROSE, D.J.M. (1997). Physical and histochemical properties of kenaf (Hibiscus cannabinus L.) grown under water deficit on a sandy soil. Ind. Crops Prod. 7, 9-18.

OKEREKE, O.O. (1962). Studies on the fibre dimensions of some Nigerian timbers and raw materials, Part 1. Res. Rep. N°16. Fed. Ministry of Commerce and Industry, Lagos, Nigeria.

PETRI, R. (1952). Pulping studies with African tropical woods. TAPPI, 35: 157-160.

RYDHOLM, S.A. (1965). Pulping process. In: Wiley and Sons (Eds.), New York, pp. 1270 (reprint from Robert E. Krieger, Malabar, Florida (facsimile edition) (1985)).

SAIKIA, S.N., GOSWAMI, T., Ali, F. (1997). Evaluation of pulp and paper making characteristics of certain fast growing plants. Wood Sci. Technol. 31, 467–475.

TISSOT, M. (1985). Papeterie Tropicale. Nogent-sur-Marne:CTFT.

VALKOMER, P.J. (1969). Wood raw materials for pulp paper in tropical countries. FAO International Review of Forestry and Forest Products; Unasylva, 23 No.3.

VERVERIS, C., GEORGHIOU, K., CHRISTODOULAKIS, N., SANTAS, P., AND SANTAS, R. (2004). Fiber dimensions lignin and cellulose content of various plant materials and their suitability for paper production. Industrial Crops and Production. 19, 245-254.

WAGENFÜHR, R. 1984. Anatomic des Holses VEB, Fachbuchuerlag Leipzig.

XU F, ZHONG, XC, SUN RC AND LU Q (2006). Anatomy, ultra structure, and lignin distribution in cell wall of caragana korshinskii. Industrial Crops & Products. 24, 186-193.

# Haptics of Wooden Flooring Elements - Influence of Temperature Sensation and Surface Roughness

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**Keywords:** wood flooring, surface roughness, temperature sensation, comfort, product design

#### ABSTRACT

The aim of the present work was to investigate factors that influence haptics and a comfortable feel of surfaces of wooden floorings with different surface properties, with a focus on oil/wax treated wood. Studies with test persons on the haptics and temperature sensation of different surfaces with hands and feet were carried out and compared to measurements of surface roughness and thermal properties. It was found that just by touching humans are able to distinguish very well between wood and synthetic surfaces and to rank surface roughness in good correspondence with measurements. A warm feel was achieved by structured surfaces like brushed larch wood while smooth wood surfaces with film forming sealers appeared cooler. The test persons considered the wood surfaces to be warmer when they could see them compared to blind assessment. Measurements with active thermography explained the reasons for warmer or cooler feel of surfaces by thermal conductivity, contact area and heat storage volume of materials.

#### **INTRODUCTION**

Besides technical criteria of wooden flooring products their haptics and temperature sensation are important factors that influence the purchase decision of end users. A good look and comfortable feel of the surface becomes more and more important to choose between alternative products. However, it is not easy to capture this human sensation and to define physical parameters that can be measured and used to improve the product's design.

Häupl (2008) described a physical model of heat flux to determine the felt surface temperature when a foot is in contact with a surface.

The contact temperature is influenced by the surface temperatures and the heat penetration coefficient of the foot and the floor. The heat penetration coefficient is determined by the heat conductivity, the density and the specific heat capacity of the materials. As humans are very much influenced by their visual perception Killian (2005) found that also the temperature sensation depends on visual factors.

Only a few studies report on haptic properties of wood surfaces. Berger et al. (2006) investigated the haptic perception mainly of laminate floorings and found that consumers are able to judge consistently the temperature, hardness and roughness of floorings just by skin contact. Teischinger et al. (2012) carried out a study on the contact comfort of wooden materials with test persons on a test chair with exchangeable seat, backrest and armrest. After 3 minutes and 10 minutes contact the main parameters creating comfort were the surface temperature, hardness and sorption activity of the surfaces. Rapp et al. (2009) studied temperature sensation depending on surface temperature of deckings heated up by sunlight using an artificial foot of latex and water. They found that surface temperature correlated with wood density and heat flux.

The aim of the present study was to investigate factors that influence haptics and a comfortable feel of surfaces of wooden floorings with different surface properties, with a special focus on oil/wax treated wood. Visual impression should be first excluded and then included in the assessment. It was the objective to find measurable parameters to characterise the haptics of these flooring elements as a basis for product design.

# MATERIAL AND METHODS

### Wood Flooring samples

Wood samples of industrially finished and unfinished flooring elements were supplied by seven Austrian producers with top layers of the wood species beech (*Fagus sylvatica*), oak (*Quercus sp.*) and larch (*Larix sp.*). The standard surfaces were planed and sanded but from larch and oak wood also brushed surfaces with rough surface structure were used. The industrial partners treated the flooring elements in their plants with different oil/wax systems (including initial care treatment) and in particular beech samples were also treated with UV-oils and UV-sealers. Other beech samples were treated manually by roller with a 1K water based acrylic sealer system for wooden floors.

A few other materials were selected as references to compare with the wood surfaces. For roughness assessment these were a metal ruler with an even

edge and two samples of high pressure laminate (HPL), one with smooth and one with rough surface. For assessing the temperature sensation a ceramic tile was used as reference.

### Studies with test persons

All assessments with test persons were carried out in conditioned rooms at  $23^{\circ}$ C and 50% relative humidity. The test persons were chosen by gender and age to gain as good as possible a representative sample.

The first study on surface roughness included 20 test persons (10 female, 10 male). In sessions with single persons they had to touch the surfaces of the samples without seeing them and to give a rating for surface roughness on a scale from 1 (smooth) to 5 (rough). To calibrate this scale they received the smoothes and the roughest sample before the test started. During the test the samples were given in a random order that was changing from one to the other test person. Together with the roughness rating the test persons had to state if they consider the surfaces to be wood or plastic.

The study on temperature sensation included 64 test persons (32 female, 32 male) evenly distributed over 5 age classes starting at 20 years. Before starting the test the test persons washed their feet and hands in water at room temperature (23°C) and dried them. In individual sessions the persons judged temperature sensation by feet and hands always in direct comparison of two different samples stating which material feels warmer than the other. The samples were chosen in random order leading to 11 comparisons and 3 control comparisons for each test person. In these comparisons the test persons ranked the temperature sensation of the samples in 4 classes from warm (rating 1) to cool (rating 4). These tests were carried out first excluding and then including the visual impression.

# High resolution profilometry

To obtain high resolution surface profiles pictures from cross sections of the samples in a Scanning Electron Microscope (SEM) that showed the real surface topography were processed. Cross sections of the surfaces of the wood flooring samples and reference materials were prepared for microscopic investigations. Non-conductive samples were sputter coated with Gold in 5 nm thickness. The cross sections were observed in a SEM (Jeol JSM-6100) and photographed with a digital imaging system at constant magnification, working distance and accelerating voltage. The pictures were calibrated to enable measurements of structure dimensions. On a length of 5.93 mm the surface profiles were traced by digital image processing as shown in

Figure 1. The profile depth was measured on these lines as distance to a straight reference line with an increment of 2.3  $\mu$ m leading to 2580 data points for each profile. Electron-optic distortion of the pictures was corrected

mathematically in order to receive straight surface profiles for the samples using the surface profile of an even edge of a metal ruler that was generated in the same procedure. The profile depth was stated as the range of data points from P1 to P99, which excluded 2 percentiles of the data with the extreme values.

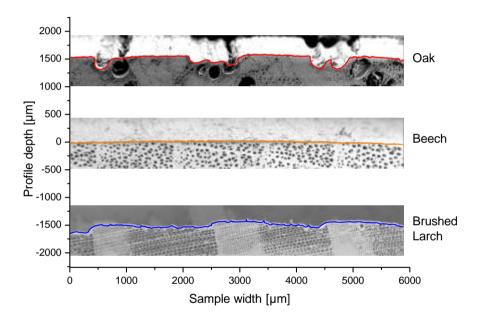


Figure 1: Digital image processing of SEM-pictures of cross sections of samples

### Active thermography

The principle of active thermography is to assess time series of thermographic pictures of an Infrared camera (IR-camera) from samples that were subjected to controlled heating. In the present study an IR-camera (Varioscan 3021 ST) was mounted on a stand in constant and parallel orientation to the test panel. This setup was placed in a conditioned room at 14°C and 50% relative humidity and covered by a cardboard box to avoid excessive air movement.

Four different samples were joined together to obtain an even surface and placed on the test panel as shown in

Figure 2. To correct differences in emissivity, one part of each sample was coated with a deep matt black laquer with known emissivity. A petri dish of glass with 93 mm diameter and an even bottom was filled with water to a height of 10 mm and heated on an electric heating mat to a temperature of approximately 55 °C. This petri dish

was placed on the sample surfaces for 30 s. After removing the petri dish the samples were photographed with the IR-camera in intervals of 4 s (Figure 2). The thermographs were processed in order to obtain mean and maximum temperature in a defined area of 30 by 30 mm centred on the heated surface of each sample.

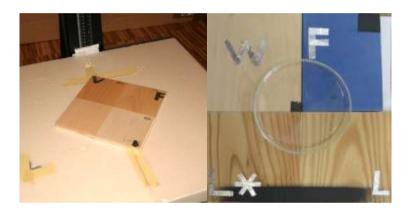


Figure 2: Four samples on test panel (left) and top view on four samples (right) with petri dish filled with water to heat the surface

# **RESULTS AND DISCUSSION**

### Surface roughness

The results of the high resolution surface profilometry are summarized as profile depth in Figure 3. The lowest profile depths were obtained from the edge of the metal ruler and the laminate references. In general the surfaces of beech wood were smoother that those of oak and larch wood. Some beech samples with oil treatment were even smoother than the untreated beech sample. The beech samples treated with sealers were relatively smooth. However, with the naked eye a difference between these two samples was visible where the surface with the UV sealer showed a characteristic wavy structure caused by the roller application that was not present at the surface with the water based sealer. From the oak samples those of the producers E and G were brushed, which explains the higher surface roughness. In general, the porous structure of oak lead to the highest values of profile depths. Also among the larch samples the brushed one was by far the roughest and only the sample from producer F was extraordinarily smooth.

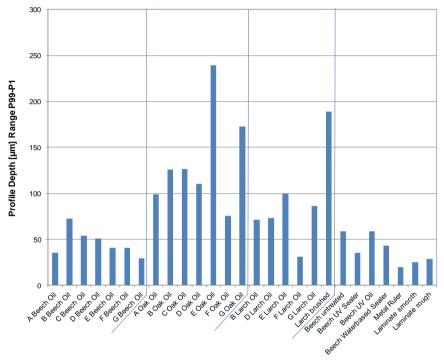


Figure 3: Surface roughness of flooring samples from producers A to G and reference materials

In the first study with test persons a relatively clear ranking of samples from very rough to very smooth was obtained as shown in Figure 4. The brush treated samples were felt as the roughest and the laminate samples as the smoothest. From the beech samples the untreated one was rated relatively rough but surprisingly the beech sample with water based sealer was found to be somewhat rougher. The beech samples with oil and UV-sealer were rated relatively smooth. In the same rank order by roughness

Figure 5 illustrates the rating of material type by the test persons. The results with untreated beech and the two laminate samples were ideal where all persons gave the correct material type. Among the other samples it was shown that those coated with sealing lacquers were more often rated as

"Hardwood Science and Technology" The 5th Conference on Hardwood Research and Utilisation in Europe 2012 plastic than the oil treated ones. These results show that humans can very precisely feel roughness and judge material type by touching a surface. With the oil/wax systems investigated the haptic character of the wood was retained very well. From the results of the samples coated with sealing lacquers it can be assumed that the temperature sensation and in this context the heat conductivity of the coatings was an important factor.

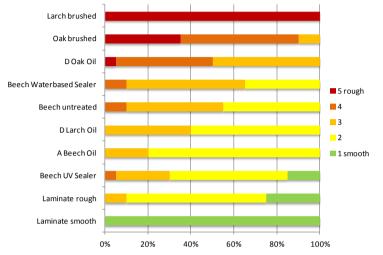


Figure 4: Rating of surface roughness by test persons (sorted from the roughest to the smoothes material)

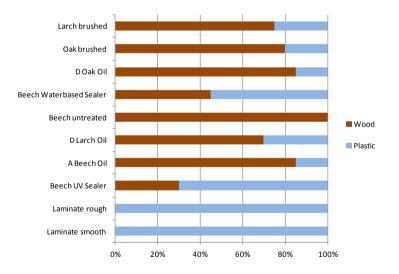


Figure 5: Statements of felt material type (sorted by roughness in the same order as in Figure 4)

Figure 6 shows that the linear correlation of the roughness rating by the test persons and the results from high resolution profilometry was relatively good. This means that the test persons were able to feel the actual surface roughness very well. It is known that humans can feel very small structures with their finger tips but it is still fascinating that this goes down to a few hundredths of a millimetre as shown in Figure 6.

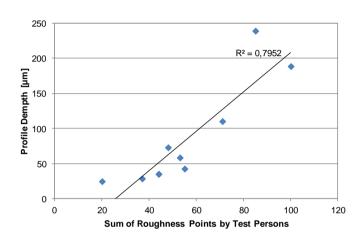


Figure 6: Correlation of roughness rating by test persons and measured roughness (profile depth)

### Temperature sensation

In the second study with test persons a general tendency was found that including the visual impression the surfaces were ranked as warmer than without visual impression (

Figure 7 and Figure 8). This was in particular the case in foot contact with the surfaces where 6 out of 8 samples were rated as warmer in the visible assessment that in the blind test (Figure 8). From the results obtained by hand contact it is interesting that only the two samples with sealers were ranked as cooler when visual impression was included. It can be concluded that the colour and structure of wood has a positive effect on temperature sensation. Surfaces with film forming coatings may have a negative influence because of the coating film and perhaps also higher gloss of the surfaces when a person is looking closely and touching by hand.

In all tests the surfaces of brushed larch wood with oil treatment were felt warmest and the coolest feeling was obtained from the surfaces of beech wood with the water based sealer. This was a consistent result in the ratings with feet and hands as well as with and without visual impression. Also the untreated beech was rated as relatively warm in all tests except the blind test by foot. Between all other samples the differences in mean values were small, which included oil treated surfaces of oak, larch and beech and the UV-curing coating systems. The film forming coating and the smooth surface of the water based sealer led to a cooler perception of the surfaces compared to others, whereas the rough surface structure of brushed larch wood and untreated beech wood appeared to be warmer.

Figure 9 gives an example of thermographs achieved by active thermography on four different samples in one trial. From these pictures it becomes evident how differently the ceramic tile (F) reacted compared to the wood surfaces. Immediately after 10 s heating the surface of the tile was heated in a smaller area that the wood samples. However, in the conditioning phase on the surface of the tile the heat spread over a larger area and it remained warmer than the other samples. The wood samples were heated in a similar pattern, where in detailed observation of the surface of brushed larch wood warmer tips of the exceeding latewood bands can be seen. In particular the larch samples with oil treatment cooled down relatively quickly, whereas the beech surface with the water based sealer cooled down slower.

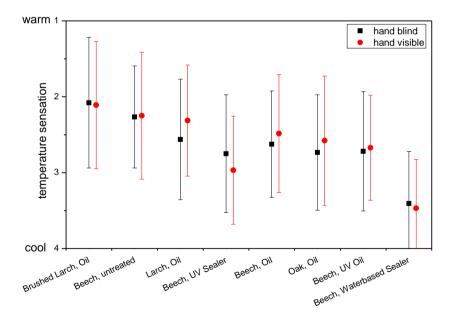


Figure 7: Rating of temperature sensation by test persons by hand excluding and including the visual impression (mean and standard deviation)

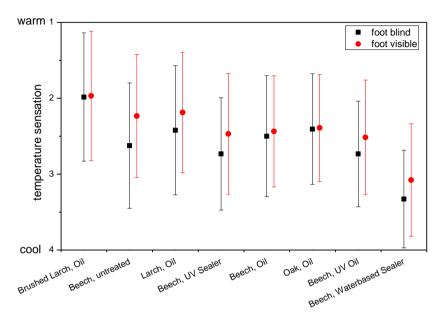


Figure 8: Rating of temperature sensation by test persons by feet excluding and including the visual impression (mean and standard deviation)

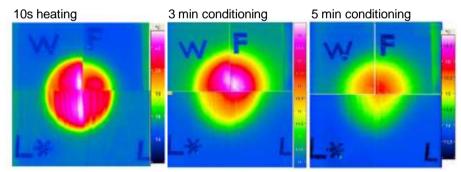


Figure 9: Thermographs of for different flooring samples (W...Waterbased sealer, F...ceramic tile, L\*...brushed larch oil treated, L...planed and sanded larch oil treated) after 10 s heating

The plot of mean temperature over the conditioning phase in Figure 10 shows equal initial temperatures on the tile and the two larch samples, while a higher temperature was found on the beech surface with the water based sealer. This can be explained by the smoother surface of the sealed beech sample leading to a larger contact area compared to the larch samples. During the conditioning phase all wood samples showed parallel curves of

mean temperature, which reflects an equal cooling rate. In contrast to this, the tile showed a smaller cooling rate. These results can be explained by the higher thermal conductivity and large heat storage volume of the tile. The wood materials on the other hand are good heat insulators, which avoid a transport of heat into the material and only the uppermost layers were heated up. The coating film of the sealer has got a higher heat conductivity as the wood but only a small storage volume because of the low film thickness of the coating (ca. 50  $\mu$ m).

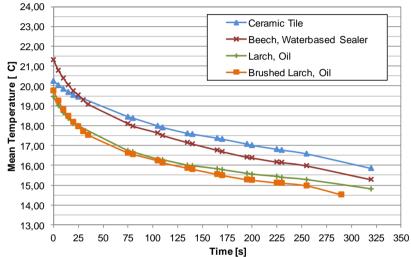


Figure 10: Mean temperature (3 independent tests) of heated sample surfaces during conditioning after 10 s heating

#### CONCLUSIONS.

Humans can very precisely feel roughness and judge material type by touching a surface. The test persons were able to feel the actual surface roughness very well which goes down to a few hundredths of a millimetre. With the oil/wax systems investigated the haptic character of the wood was retained very well. From the ratings of test persons on the samples coated with sealing lacquers it can be assumed that the temperature sensation and in this context the heat conductivity of the coatings is an important factor.

It was found that the colour and structure of wood has a positive effect on temperature sensation. Surfaces with film forming coatings may have a negative influence because of the coating film and perhaps also higher gloss of the surfaces when a person is looking closely and touching by hand. The film forming coating and the smooth surface of the water based sealer led to a cooler perception of the surfaces compared to others, whereas the rough surface structure of brushed larch wood and untreated beech wood appeared to be warmer.

The reasons for warmer or cooler feel of surfaces are thermal conductivity of the material, contact area with the surface (surface roughness) and heat storage volume of materials. The low heat conductivity of wood is beneficial for a warm feel of wood surfaces because the uppermost layers are heated up quickly by the temperature of the human body.

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### REFERENCES

Berger G., Katz H., Petutschnigg A. (2006): What consumers feel and prefer: Haptic

perception of various wood flooring surfaces. Forest Products Journal 56, 10, 42-47

HÄUPL P. (2008): Bauphysik – Klima Wärme Feuchte Schall. Grundlagen, Anwenwendungen Beispiele, Ernst Wilhelm & Sohn, Berlin

KILLIAN K.; BREXENDORF O. (2005): Multisensuale Markenführung als Differenzierungs- und Erfolgsgröße, Campus02 Business Report, Vol. 2: 12-15.

RAPP, A.O.; WELZBACHER, C.R.; BRISCHKE, C. (2009); Barefoot-heatimpact of oil-heat-treated wood - An important thermal property of decking boards, Leibniz University Hannover (LUH), Faculty for Architecture and Landscape Sciences, Institute of Vocational Sciences in the Building Trade

TEISCHINGER A, ZUKAL M L, KOTRADYOVA V (2012): Exploring the possibilities of increasing comfort by wooden materials - Tactile interaction f man and wood. Innovation in Woodworking Industry and Engineering Design 1, 1/2012, 20-24.

# Variation in Wood Fiber Characteristics Among Thirty Two Hardwood Species Grown in Low-Rainfall Wood land Savannah (Sudan)

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Keywords: Variation, Fiber characteristics, Hardwood species.

#### ABSTRACT

Sudan is endowed by a great variation of climatic zones with an annual rainfall extending from less than 75 mm in deserts to over 1500 mm in the mountainous regions. With The great variation on the climatic zones of Sudan great variations are expected in the anatomical properties between and within species. Previous studies have shown that fiber morphology is an important indicator for wood end-use as it strongly affects on the general quality of most of the products produced from wood. Wood fiber differs in its characteristics from species to another. This variation need to be fully explored in order to suggest best uses for the species.

The present study aimed to investigate the variation on wood fiber characteristics among some hardwood species growing in low rainfall woodland savannah of Sudan. This study aimed also to provide good bases for wood fiber characteristics of the selected species in order to suggest best uses for them. Wood materials, from thirty two hardwood species belonging to eighteen families, collected from Southern Kordofan state and Sennar state were used for this purpose. Several fiber characteristics were investigated. These are length, diameter, lumen diameter, wall thickness, slenderness ratio, Runkle ratio and flexibility coefficient.

The analysis of variance and Duncan's Multiple Range Test were conducted to study the\_significance of variation among species and to separate means using SAS GLM procedure. The results showed highly significant variation among species in all the investigated fiber characteristics. Generally, the investigated wood fiber dimensions of the studied species were in the normal rang for hardwood species. The current study results promote the possibility of using the study species on forest industries.

# INTRODUCTION

Sudan has a wide variation of climatic zones with an annual rainfall extending from less than 75 mm in deserts to over 1500 mm in the mountainous regions. This variation in climatic zones has a direct impact on the immense diversity and variation in the vegetation of the country. With the great variation on the climatic zones of Sudan, great variations are expected in the anatomical properties between species. Understanding the extent of variability of wood is important because the uses for each kind of wood are related to its characteristics (PANSHIN AND DE ZEEUW 1980).

Wood, like all plant materials, is made up of cells of different shapes and sizes. The mechanical support in wood of hardwoods is due to long and narrow cells with closed ends namely fibers (PANSHIN AND DE ZEEUW; 1980; TSOUMIS 1968; JANE 1970 AND ILVESSALO-PFÄFFLI 1995). The fiber, as one of the structures of wood, differs in its characteristics (e.g. length, diameter, shape, etc.) from one species to anther (PANSHIN AND ZERUW, 1980). These variation leads to variation on the possible end uses for each species.

Previous studies have shown that fiber morphology is an important indicator for end-use. For instance it affects papermaking properties of wood species. Some researchers like SAIKIA ET AL. (1997), OGBONNAYA ET AL. (1997) and VERVERIS ET AL. (2004) have successfully used the fiber derived values to assess the suitability of various fiber raw materials for pulp and paper manufacture. RASHEED AND DASTI (2003) reported that the shape of fiber cell, its length and wall structure are important in the fiber industry. Fiber length affects the strength, surface, and bonding properties of fiber products and is therefore of interest, for many purposes, long fibers are more desirable than short ones (DADSWELL AND NICHOLLS 1959). Long fibers shrink less longitudinally than short fibers. For some purposes, short fibers are required, either alone or in mixture with long fibers (KOCH 1985). Mechanical timber strength is related closely to its weight; more accurately, to its density or weight per unit volume and this in turn depends largely on the proportion of fiber cells in its make-up and the thickness of their cell walls (WILSON AND WHITE 1986). Generally, higher proportion of thick-walled fibers is associated with higher strength (HAYGREEN AND BOWYER 1989, BUES AND KÖNIG 2004).

Very meagre information is available on fiber characteristics of wood species growing in Sudan. Sudan, even in the low rainfall savannah, is rich in wood species belonging to different taxa and the wood structure is species-specific; this is expected to lead to great variations in fiber characteristics. Fiber characteristics can be used as a good guide for suitable uses of wood. The study of fiber characteristics is expected to give a fundamental basis for fiber classification and wood identification.

This work was an attempt to shed some lights on the variations in fiber characteristics of thirty two hardwood species belonging to eighteen families grown in the low rainfall woodland savannah of Sudan.

## MATERIALS AND METHODS

### Materials

Wood material was collected from thirty-two hardwood tree species belonging to eighteen families. The species were growing in low-rainfall savannah, in five forests namely, Al-Dalang, Al Debaibat, Al Tokma, Al-Faid Um Abdalla (which are located in Southern Kordofan State) and Al-Lambowa Forest (which is located in Sennar State). Table no 1 represent the families, scientific names of the selected tree species.

Family	Scientific Name		
Anacardiaceae	Lannea schimperi		
	Sclerocarya birrea		
Burseraceae	Boswellia papyrifera		
	Cassia fistula		
Caesalpiniaceae	Delonix regia		
	Tamarindus indica		
	Anogeissus leiocarpus		
Combertaceae	Combretum hartmannianum		
	Terminalia brownii		
Ebenaceae	Diospyros mespiliformis		
Euphorbiaceae	Euphorbia tirucalli		
Malvaceae	Adansonia digitata		
Walvaccac	Ceiba pentandra		
	Azadirachta indica		
Meliaceae	Khaya senegalensis		
	Acacia gerrardii		
	A.senegal		
Mimosaceae	A.seyal		
	A.sieberana		
	A. nilotica		
	Albizia amara		
Moraceae	Ficus sycomorus		
Myrtaceae	Eucalyptus camaldulensis		
	Dalbergia melanoxylon		
Papilionaceae	D. sisso		
Rhmnaceae	Ziziphus spina-christi		
Simaroubaceae	Ailanthus excelsa		
Sterculiaceae	Sterculia setigera		
Tamaricaceae	Tamarix aphylla		
Verbenaceae	Gmelina arborea		
	Tectona grandis		
Zygophyllaceae	Balanites aegyptiaca		

Table 1: List of families, scientific names of the study species

Samples of Acacia nilotica, A.seyal var. seyal, Tamarix aphylla, Ficus sycomorus and Ziziphus spina-christi species were collected from Al-Lambowa Forest. Samples of Acacia senegal, A. gerrardii, Ailanthus excelsa, Azadirachta indica, Balanites aegyptiaca, Cassia fistula, Diospyros mespiliformis, Khaya senegalensis and Euphorbia tirucalli species were collected from Al-Dalang Forest, while samples of the remaining species were collected from Al-Faid Oum Abdala Forest. Three healthy mature trees were randomly taken from each species. From each tree, two representative samples were obtained from the outer wood of the stem at breast height from opposite sides of the tree using an increment borer.

## Methods

# Maceration Procedure

The maceration procedure developed by Shultze as cited in JANE (1970) was used to macerate the woody materials into individual cells using a strong agent (nitric acid 60%) that breaks down the less resistant middle lamellae so that the elements fall apart and could be studied individually as solid structures.

Small slivers of wood were placed in a test-tube, to which 60% nitric acid was added and then warmed up in a water bath. Depending upon the wood species, the time of warming (maceration) was 5-15 minutes. The macerated material was washed several times by distilled water to remove traces of nitric acid and then left for about 10 minutes in distilled water. When the loosened sections settled down, excess water was gently drained away, the macerated materials were placed in Petri dishes, washed in 25 and then 50% alcohol (ethanol), stained by few drops of safranin dye and then rewashed using a series of alcohol concentrations (50%, 70%, and 95%). The prepared macerated materials were placed in slides and one drop of DPX was added to each slide. Each slide was then covered gently with a cover slips and left to dry gradually for a day.

# **Microscopic examination**

Quantitative examinations were carried out, these included fibers length, fibers diameter, and fibers lumen diameter. All of these characteristics were measured using a light microscope (model: hund WETZLAR) with a 10x ocular lens provided with a measuring scale graduated into ten equal segments and each segment is then graduated into ten sub-segments. Fibers length was measured under 10x objective lens, while fibers diameter and fibers lumen diameter were measured under40x objective lens.

For each character examined, twenty fibers were randomly measured from each sample giving a total of 120 measurements per species (20 fibers per sample x 2 samples per tree x 3 trees per species). The measured values were transformed into real values by calibrating the measuring-scale using a calibration scale (one millimeter). Fibers wall thickness was calculated using the following equation:

$$FWT = \frac{FD - FLD}{2} \tag{1}$$

Where: FWT is double wall thickness; FD is diameter and FLD is lumen diameter

The obtained fiber dimensions values were used to calculate the slenderness ratio (Eq 2), Runkel ratio (Eq 3) and flexibility coefficient of suppleess (Eq 4 as follow:

$$SR = \frac{FL}{FD} \tag{2}$$

$$RR = \frac{2FWT}{FLD} \tag{3}$$

$$FC = \frac{FLD}{FD} \times 100 \tag{4}$$

Where: SR = Slenderness ratio RR = Runkel ratio FC = Flexibility coefficient FL = Fiber length (µm) FD= Fiber diameter (µm)

### **Statistical Analysis**

SAS procedures were used to study the variation in wood fiber characteristics. The analysis of variance and Duncan's Multiple Range Test were conducted to study the significance of variation among species and to separate means using SAS GLM procedure.

### **RESULTS AND DISCUSSION**

The results of the current study showed highly significant variation among species in all the investigated fiber characteristics. Table no 2 represents the wood fiber characteristics mean values of the study species.

Species	Fiber Dimensions [µm]				Fiber Derived Values*		
	Length [mm]	Diameter	Lumen diameter	Wall thickness	FC	RR	SR
Acacia gerrardii	1.19	20.0	6.28	6.85	42	2.60	61.5
A. nilotica	1.02	18.6	7.23	5.70	39	1.88	54.7
A.senegal	1.02	17.3	7.28	5.01	42	1.71	62.0
A.seyal var.seyal	1.09	18.8	7.87	5.49	42	1.65	59.9
A.sieberana	1.20	21.5	7.62	6.97	35	2.30	56.9
Adansonia digitata	2.84	34.0	24.24	4.90	70	0.50	89.1
Ailanthus excelsa	1.10	29.7	22.70	3.51	76	0.34	35.1
Albizia amara	0.99	19.6	10.04	4.77	51	1.03	51.1
Anogeissus leiocarpus	1.01	14.2	4.78	4.69	34	2.14	70.8
Azadirachta indica	0.83	19.5	10.27	4.61	52	1.02	42.7
Balanites aegyptiaca	1.04	20.6	10.04	5.29	49	1.14	51.9
Boswellia papyrifera	0.91	32.9	22.56	5.18	68	0.50	28.1
Cassi fistula	0.93	17.6	7.31	5.08	42	1.59	50.8
Ceiba pentandra	2.41	32.4	24.12	4.13	74	0.38	76.3
Combretum hartmannianum	0.89	18.6	6.99	5.81	38	1.92	50.2
Dalbergia melanoxylon	0.56	17.2	7.06	5.06	41	1.63	33.2
D. sisso	0.99	20.1	11.93	4.09	59	0.78	51.8
Delonix regia	1.11	33.1	25.35	3.87	76	0.34	35.2
Diospyros mespiliformis	0.98	18.8	9.08	4.86	48	1.21	50.2
Eucalyptus camaldulensis	0.76	17.3	12.28	3.64	63	0.65	44.1
Euphorbia tirucalli	0.83	21.0	13.97	3.51	66	0.58	40.8
Ficus sycomorus	1.27	24.0	15.69	4.28	64	0.61	54.8
Gmelina arborea	1.13	28.6	22.88	2.88	80	0.27	39.5
Khaya senegalensis	1.19	19.3	9.85	4.74	51	1.08	57.5
Lannea schimperi	0.93	33.7	24.94	4.38	73	0.45	28.1
Sclerocarya birrea	0.97	28.4	20.01	4.21	70	0.48	35.2
Sterculia setigera	1.95	25.5	12.55	6.32	49	1.13	75.8
Tamarindus indica	0.82	17.8	9.03	4.39	51	1.08	46.1
Tamarix aphylla	0.75	20.9	13.01	3.89	63	0.65	36.7
Tectona grandis	1.01	27.3	20.81	3.24	76	0.40	38.8
Terminalia brownii	1.06	17.1	7.04	5.01	41	1.66	62.0
Ziziphus spina-christi	1.02	19.1	11.16	3.92	57	0.77	57.7

Table 2: Fiber characteristics mean values of the study species

\*FC = Flexibility coefficient, RR = Runkel ration and SR = Slenderness ratio

In agreement with the significant difference in fiber length among species were those reported by MANWILLER (1974), YOUSSIF (2000), OSMAN (2001), RASHEED AND DASTI (2003). However, the results of this study were in disagreement with BABOS (1979) who didn't report significant variation among species. *Adansonia digitata* had significantly the highest value of fiber length with a mean of 2.84 mm, followed by *Ceiba pentandra* with a mean of 2. 41 mm and then by *Sterculia setiger* with a mean of 1.95  $\mu$ m. Significantly lower mean value of fiber length (0.561mm) was associated with *Dalbergia melanoxylon* compared to the other species.

Fiber length of the studied species had been classified according to IAWA COMMITTEE (1989) and WAGENFÜHR (1984) classification as shown in Table 3.

Species	Mean values	Classes*		
	[mm]	IAWA	Wagenführ	
		(1989)	(1984)	
Adansonia digitata	2.84	L	L	
Ceiba pentandra	2.41	L	L	
Sterculia setigera	1.95	L	М	
Ficus sycomorus	1.27	М	S	
Acacia sieberana	1.12	М	S	
Khaya senegalensis	1.19	М	S	
Acacia gerrardii	1.19	М	S	
Gmelina arborea	1.13	М	S	
Delonix regia	1.11	М	S	
Ailanthus excels	1.11	М	S	
Acacia seyal var.seyal	1.09	М	S	
Terminalia brownii	1.06	М	S	
Acacia Senegal	1.04	М	S	
Balanites aegyptiaca	1.04	М	S	
Ziziphus spina-christi	1.02	М	S	
Acacia nilotica	1.02	М	S	
Tectona grandis	1.01	М	S	
Anogeissus leiocarpus	1.01	М	S	
Dalbergia sisso	0.989	М	VS	
Albizia amara	0.985	М	VS	
Sclerocarya birrea	0.969	М	VS	
Diospyros mespiliformis	0.975	М	VS	
Cassia fistula	0.929	М	VS	
Lannea schimperi	0.931	М	VS	
Boswellia papyrifera	0.912	М	VS	
Combretum hartmannianum	0.894	S	VS	
Azadirachta indica	0.834	S	VS	
Euphorbia tirucalli	0.832	S	VS	
Tamarindus indica	0.821	S	VS	
Eucalyptus camaldulensis	0.756	S	VS	
Tamarix aphylla	0.749	ŝ	VS	
Dalbergia melanoxylon	0.561	S	VS	

 Table 3: Fiber length classification according to IAWA (1989) and Wagenführ (1984)

\*L = Long, M = Midium, S = Short and VS = Very short

The fiber diameter significant variations detected in the current study are in agreement with those found by OSMAN (2001) and RASHEED AND DASTI (2003). The highest mean values of fiber diameter were recorded for *Adansonia digitata* (34  $\mu$ m), while the lowest mean value was found in *Anogeissus leiocarpus* (14.2  $\mu$ m).

The study results are in accordance with OSMAN (2001), RASHEED AND DASTI (2003) who observed significant variation in fiber lumen diameter among species. The values of mean fiber lumen diameter of the studied species ranged from 4.8 µm in *Anogeissus leiocarpus* to 25.4 µm in *Delonix regia*. Concerning the fiber wall thickness, the significantly differences found in this study are in agreement with those of OSMAN (2001), but different than those of BABOS (1979) and RASHEED AND DASTI (2003) in which they didn't find significant variation among species. The values of

wall thickness varied from 2.8 µm in *Gmelina arborea* to 6.96 µm in *Acacia sieberana*.

In contrast to the slenderness ratio significantly differences found in current study, RASHEED AND DASTI (2003) didn't find significant variation in slenderness ratio among twenty four plant species belonging to nineteen families. The highest mean value of slenderness ratio (89.1) was associated with *Adansonia digitata* and the lowest value (28) was associated with *Boswellia papyrifera*.

In disagreement with the current study results RASHEED AND DASTI (2003) reported no significant variation in the values of runkle ratio among twenty four monocot and dicot species. *Acacia gerrardii* had significantly the highest mean value of runkle ratio (2.6), while *Gmelina arborea* had the lowest mean value (0.27). The flexibility coefficient significant variations detected in the current study are in agreement with those found by RASHEED AND DASTI (2003). The highest flexibility coefficient mean value (0.80) was in *Gmelina arborea* while the lowest mean value was in *Acacia gerrardii* (0.32).

### CONCLUSION

Highly significant variations were found among species in all the investigated fiber characteristics. These variation leads to expect variation also in their end uses. Some of the study species have long fibers which are comparable with those of softwood. Almost all the study species have good slenderness ratio (> 33). Runkel ratios of more than twenty species are within the acceptable range for pulp and paper making of 0.25-1.5 as sited by VALKOMER (1969). While half the study species have values of more than 60 % flexibility coefficient which is acceptable for pulp and paper making as sited by (PETRI 1952, OKEREKE 1962 AND RYDHOLM 1965). The current study results promote the possibility of using the study species on forest industries.

### REFERENCES

BABOS, K. (1979). Fiber characteristics of some cuban hardwoods by karoly babos. Research institute for the wood working industry, vorosmarty. 56,H-1201 Budapest, Hungary.

BUES, C.T. AND KÖNIG, J. (2004). Properties and Utilization of Tropical Hardwoods and Softwoods from Plantations, for Postgraduate Degree Advanced Academic Course (MSc) Tropical Forestry and Management. Dresden Univesity.

DADSWELL, H.E. AND NICHOLLS, J.W.P. (1959). Assessment of wood qualities for tree breeding.1.CSIRO Div. *Forest Products Technology* Pap. 4,16p.

HAYGREEN, J.G. AND BOWYER, J.L. (1989). Forest Products and Wood Science. An introduction 2<sup>nd</sup> ed. IOWA State University Press, Ames, 500.

IAWA COMMITTEE. (1989). IAWA list of microscopic features for hardwood identification. *IAWA Bull. n.s* 10 (3): 221-332.

ILVESSALO-PFÄFFLI, M.S. (1995). Fiber Atlas, identification of papermaking fibers. Springer Series in Wood Science. Editor T.E. Timell.

JANE, F.W. (1970). The Structure of Wood. 2<sup>nd</sup> ed. Completely revised by K. Wilson and D.J.B White. London.

KOCH, P. (1985). Utilization of Hardwoods Growing on Southern Pine Sites. Agriculture handbook number 605. U.S. Department of agriculture. Forest service. Libarary of Congress.

MANWILLER, F.G. (1974). Fiber lengths in stems and branches of small hardwoods on southern pine sites. *Wood Science* 7:130-132.

OGBONNAYA, C.I., ROY-MACAULEY, H., NWALOZIE, M.C., AND ANNEROSE, D.J.M. (1997). Physical and histochemical properties of kenaf (Hibiscus cannabinus L.) grown under water deficit on a sandy soil. Ind. Crops Prod. 7, 9-18.

OKEREKE, O.O. (1962). Studies on the fibre dimensions of some Nigerian timbers and raw materials, Part 1. Res. Rep. N°16. Fed. Ministry of Commerce and Industry, Lagos, Nigeria.

OSMAN, M.E.(2001). Variation in density and fiber dimensions and the dimarcation of juvenile and mature wood of ten tree species grown in Sudan. M.Sc. Thesis, University of Khartoum, Sudan.

PANSHIN, A.J. AND DE ZEEUW, C. (1980). Textbook of Wood Technology 4<sup>th</sup> ed. Mc Graw-Hill Book Company. New York.

PETRI, R. (1952). Pulping studies with African tropical woods. TAPPI, 35: 157-160.

RASHEED, S. and A.A. DASTI. 2003. Quality and Mechanical Properties of Plant commercial Fibers. *Pakistan Journal of Biological Sciences* 6(9): 840-843. http://www.ansinet.org/fulltext/pjbs/pjbs69840-843.pdf.

RYDHOLM, S.A. (1965). Pulping process. In: Wiley and Sons (Eds.), New York, pp. 1270 (reprint from Robert E. Krieger, Malabar, Florida (facsimile edition) (1985)).

SAIKIA, S.N., GOSWAMI, T., AND ALI, F. (1997). Evaluation of pulp and paper making characteristics of certain fast growing plants. Wood Sci. Technol. 31, 467–475.

TSOUMIS, G. (1968). Wood as Raw Material: source, structure, chemical composition, growth, degradation and identification. Pergaman Pres. Oxford. London. Edinburgh. New York. Toronto. Sydney. Paris. Braunschweing.

VALKOMER, P.J. (1969). Wood raw materials for pulp paper in tropical countries. FAO International Review of Forestry and Forest Products; Unasylva, 23 No.3.

VERVERIS, C., GEORGHIOU, K., CHRISTODOULAKIS, N., SANTAS, P., AND SANTAS, R. (2004). Fiber dimensions lignin and cellulose content of various plant materials and their suitability for paper production. Industrial Crops and Production. 19, 245-254.

WAGENFÜHR, R. (1984). Anatomic des Holses VEB, Fachbuchuerlag Leipzig.

WILSON, K. AND WHITE, D.J.B. (1986). The Anatomy of Wood: its Diversity and Variability. Stobart and son ltd London.

YOUSIF, S.M. (2000). Comparative wood anatomy of some *Acacia* Species in blue nile region, Central Sudan. M.Sc. Thesis, University of Khartoum, Sudan.

# Densification of beech wood: Furfuryl alcohol impregnation for improved plasticization, fixation and properties

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**Keywords:** Compressed beech wood, furfuryl alcohol, hardness, springback-effect

### ABSTRACT

The well-known densification of wood leads to better mechanical properties, e.g., an increased hardness and density. However, in contact with water densified wood almost returns back into its original shape. Hence the aim of this study was the evaluation of a combined densification and fixation process. Beech wood samples (*Fagus sylvatica* L.) were impregnated with a solution consisting of furfuryl alcohol, maleic anhydride and ethanol. The compression of these samples to approximately 30% followed by a curing process in a heating press resulted in a significant increase of hardness and dimensional stability and significant reduced shaping forces. The spring-back-effect was clearly reduced by the in situ polymerization of the furfuryl alcohol solution to furan resin. It was also shown that the furfurylalcohol reduces the microscopic defects which occur during the compression of native wood.

#### **INTRODUCTION**

Compression is one method to improve the mechanical properties of wooden materials. Seborg et al. (1945) first reported that solid wood can be compressed. Exposed to moisture, untreated compressed solid wood tends to

undergo an irreversible spring-back-effect (Navi and Girardet 2000). Stabilization the densified shape by various combinations of compressive, thermal and chemical treatments has been the purpose of many studies (Kamke 2006). Thereby the compression process itself has been improved (Blomberg and Persson 2004; Blomberg et al. 2006) or the compressed wood has been stabilized by heat treatment or steaming (Inoue et al. 1993a, 2008). Dwianto et al. (1997), Boonstra and Blomberg (2007), Welzbacher et al. (2008) have evaluated the permanent fixation of compressed wood by heat treatment. Navi and Girardet (2000) have developed a thermo-hygromechanical treatment to obtain stable deformations. Nevertheless, the reduction in wood strength is the main problem of processes at high temperatures (Inoue et al. 1993a; Dwianto et al. 1997).

The method of filling the void wood volume with resin and subsequently compression (Vorreiter 1934; Shams et al. 2004; Gabrielli and Kamke 2010) of wood or veneer (Stamm and Seborg 1939; Kamke 2006) is another approach to reduce the spring-back-effect. This process leads to solidification caused by chemical reactions and a reduced water uptake duo to solid polymer in the vessels and the cellwall. It is well known that furfuryl alcohol is a reactive component for new wood adhesives (Kim et al. 1994; Dao and Zavarin 1996; Gosselink et al. 2011). The interaction of furfuryl alcohol with wood was first mentioned by Stamm's students Goldstein and Dreher (Goldstein 1955, Goldstein & Dreher 1960). They reported a high dimensional stability and an improved decay resistance but problems with the used zink-chloride catalyst system never led to a bigger production of furfurylated wood. In the 1990s Schneider and Westin developed new catalyst systems which is soluble in water or alcohol (Schneider 1995, Westin 1996). Under Schneiders supervision these systems led to the Kebony process and the production of furfurylated wood with several improved properties for cladding, decking and other applications (Lande 2004). In the present study, a new, compressed wood polymer composite was developed based on a combined modification with furfuryl alcohol and an densification process. The hypothesis was that furfuryl alcohol enhances plasticization and fixation at elevated temperature and that this reagent is forming a furan resin in the compressed wood due to an acid-catalysed polymerization of the furfurylalcohol.

### **EXPERIMENTAL METHODS**

Each test series comprised eight specimens (40x40x15 mm3, LxTxL) of beech (*Fagus sylvatica* L.) taken from the same plank. A mixture of furfuryl alcohol [technical grade, 97%, supplied by Fluka (Fluka Analytical,

St. Louis, Missouri, USA)] and ethanol in varying ratios was used as impregnation solution. Five percent (w/w) maleic anhydride (based on the furfuryl alcohol solution) was added as catalyst. Different weight percentage gains (WPGs) have been obtained by means of different ratios of ethanol (0%, 25%, 50% and 75%) as described by Hadi et al. (2005). Vacuum/pressure impregnation was performed in an autoclave [0.1 bar for 30 min followed by 4.5 bar for 90 min; Lande et al. (2004) and Venas and Felby (2009)]. The samples were stored in a plastic bag for 24 h after impregnation to ensure that full swelling occurred after penetration of the cell wall with furfuryl alcohol. After vaporizing ethanol at 40°C, the impregnated samples were densified in radial direction at temperature of 120°C. A compression set c of 30% was realised:

$$C = \frac{R_0 - R_c}{R_0} \times 100 \, [\%] \tag{1}$$

where  $R_0$  and  $R_c$  are the sizes in the compressed direction at room temperature before, and after, compression. Stress-strain curves were recorded during the compression process for characterizing the compression behavior. The samples remained in the closed press for 30 min, allowing the polymerization process to start. Afterwards, the samples were stored in a laboratory oven at 103°C for 24 h (post-curing step). This step ensured that the polymerization process was completed and unpolymerized reactants were removed. The oven dry density (DIN EN 13183-1) and the Brinell hardness (DIN EN 1534) were determined. Subsequently, a cyclic water storage and drying process was conducted to evaluate the spring-backeffect according to Inoue et al. (1993b). For that purpose, the ovendried samples were exposed to a 0.1 bar vacuum for 30 minutes. Then, 20°C warm water was added to the samples for 24 hours. Afterwards, the samples were oven dried again. This procedure was repeated three times. The spring-backeffect,  $R_s$ , was determined after the third cycle:

$$R_{S} = \frac{l_{r} - l_{c}}{l_{0} - l_{c}} \times 100 \, [\%]$$
<sup>(2)</sup>

where  $l_r$  is the radial thickness under oven-dried conditions after the third water storage and  $l_0$  and  $l_c$  are the oven-dried radial thicknesses before and after compression.

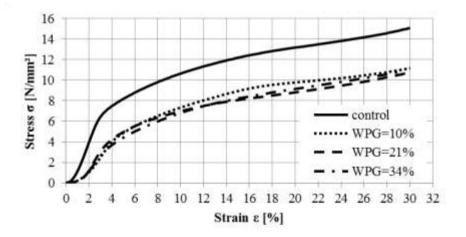
For a better understanding of the behaviour of the cell structure due to compression and springback, microscopic pictures were taken.

Therefore furfurylalcohol impregnated and native densified wood was compared before and after the cyclic water storage test. The samples were prepared for incident light microscopy by embedding them in epoxy resin and polishing the surface.

The dimension stability, ASE, was measured as the total volume swelling coefficient of the impregnated and densified samples  $S_m$  compared to native densified samples,  $S_u$  after the third wetting cycle:

$$ASE = \frac{S_u - S_m}{S_u} [\%]$$
(3)

The control samples for all tests have been oven dried and densified (c=30%) without impregnation or just ovendried.



# **RESULTS AND DISCUSSION**

Figure 1: Stress-strain curves by densification of unmodified and furfuryl alcohol (FA) impregnated beech samples (densification ratio: 30%).

The impregnation with furfuryl alcohol results in a significant plasticization of the wood. Stress strain curves of a reference native and furfuryl alcohol-treated samples are shown in Figure 1 (c=30%). The furfuryl alcohol clearly reduces the applied deformation forces compared to native wood.

Thereby this effect is not depending on the amount of furfurylalcohol between 10% and 43% weight per gain, which is equal to 25% furfurylalcohol and 100% furfurylalcohol in the treating solution. A furfurylalcohol concentration of more than 25% in the treating solution does not lead to any further improvement of the plasticization during the densification.

Young's modulus and yield stresses are reduced by 50% (Table 1).

pregnation with furfuryl alconol (FA) (medians, standard deviations in parentnes						
		MOE	Yield stress			
		[N/mm <sup>2</sup> ]	[N/mm <sup>2</sup> ]			
	Control	305 (21)	7.7 (0.3)			
	25% FA (WPG=10%)	154 (5)	4.4 (0.1)			
	50% FA (WPG=21%)	163 (22)	3.8 (0.3)			
	100% FA (WPG=34%)	142 (10)	3.7 (0.1)			

 Table 1: Reduction of Young's modulus MOE and yield stress during compression after

 impregnation with furfuryl alcohol (FA) (medians, standard deviations in parentheses).

The densitiy and the brinell hardness in dependence of the WPG which are reached with c=30% are presented in table 2. It can be seen, that the major increase in density is reached due to the densification. In addition the density rises with increasing WPG. The brinell hardness also behaves in this manner. Densification of 100% furfurylalcohol impregnated beech wood to c=30% improves the brinell hardness up to 270%.

 Table 2: Density and brinell hardness after densification and curing. (medians, standard deviations in parentheses)

	Density	Brinellhardness
	[kg/m <sup>3</sup> ]	[N/mm <sup>2</sup> ]
Control	734 (4)	49 (4)
Control, c=30%	913 (4)	106 (4)
25% FA (WPG=10%)	932 (3)	92 (6)
50% FA (WPG=21%)	937(3)	124 (7)
100% FA (WPG=34%)	1062 (7)	133 (7)

The spring-back is clearly reduced with increasing WPG (Fig. 2). Accordingly, the furan resin successfully fixes the wood in the compressed state.

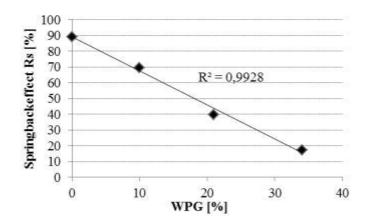


Figure 2: Spring-back-effect of furfuryl alcohol modified and densified beech wood samples after three drying-wetting-cycles.

The dimensional stability of the modified and densified wood also clearly increases (Fig. 3).

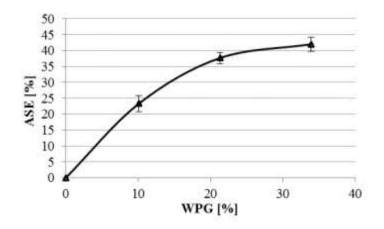


Figure 3: Dimensional stabilisation (ASE) relating to native densified samples after the third wetting cycle, c=30%.

The possibility for water to bind on free hydroxyl groups is decreasing with increasing WPG. Thereby a WPG of more than 25% does not increase the ASE much more, because the cellwalls are full of furan resin and further amounts of furan resins will deposit in the lumen which has no influence on the swelling behaviour.

The microscopic images are shown below. The densification of native beech wood to c=30% leads to highly deformed rays, vessels and libriform fibres. The lumen of the vessels is still open, several lumen of the librifoem fibres are closed due to compression (Fig. 4). The vessels and parts of some rays are showing some serious cracks. In opposite the furfurylalcohol leads to a better plasticization. Rays, vessels and libriform fibres in furfurylalcohol treated and densified wood are deformed aswell, but there is no obvious damage of the whole structure (Fig. 7). The wetting cycle inducing the spring back leads to an permanent reshaping of vessels and libriform fibres. The lumen is opening up, the shape of the vessels is almost as before compression. Native wood shows damaged vessels and cracks in the rays (Fig. 7). The springback is not damaging vessels and libriform fibres due to the bounded furan resin in the wood structure.

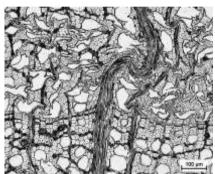


Figure 4: Native densified beech wood, c=30% (100x)

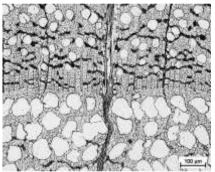


Figure 5: Native densified beech wood ( c=30%) after the third drying wetting cycle (100x)

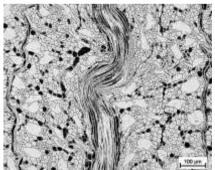


Figure 6: 50% furfurylalcohol treated densified beech wood, c=30%, WPG 21% (100x)

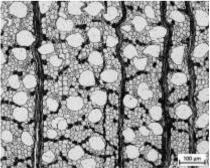


Figure 7: Furfurylalcohol treated densified beech wood (c=30%, WPG 21%) after the third drying wetting cycle (100x)

### CONCLUSION

Furfuryl alcohol causes a plasticization as well as a shape fixation in a combined densification and shaping process. The compression of impregnated samples at approximately 30% and following curing in a heating press results in a increase in hardness, density and dimensional stabilization after the third wetting cycle. The spring-back-effect can be clearly reduced compared with native densified wood. The furfuryl alcohol is able to preserve the cell structure from damages due to the compression process even after wetting and drying such modified wood several times.

### REFERENCES

BLOMBERG, J., PERSSON, B. (2004) Plastic deformation in small clearpieces of Scots pine (Pinus sylvestris) during densification with the CaLignum process. *J. Wood Sci.*, **50**, 307–331.

BLOMBERG, J., PERSSON, B., BEXELL, U. (2006) Effects of semiisostatic densification on anatomy and cell-shape recovery on soaking. *Holzforschung*, **60**,322–331.

BOONSTRA, M., BLOMBERG, J. (2007) Semi-isostatic densification of heat-treated radiata pine. *Wood Sci. Technol.*, **41**, 607–617.

DAO, L.T., ZAVARIN, E. (1996) Chemically activated furfuryl alcoholbased wood adhesives. I. The role of furfuryl alcohol. *Holzforschung*, **50**, 470–476.

DIN EN 13183-1:2002 Feuchtegehalt eines Stückes Schnittholz - Teil 1: Bestimmung durch Darrverfahren.

DIN EN 1534:2010 Holzfußböden – Bestimmung des Eindruckwiderstands – Prüfmethode.

DWIANTO, W., INOUE, M., NORIMOTO, M. (1997) Fixation of compressive deformation of wood by heat treatment. *Mokuzai Gakkaishi*, **43**, 303–309.

GABRIELLI, C., KAMKE, F.A. (2010) Phenol–formaldehyde impregnation of densified wood for improved dimensional stability. *Wood Sci. Technol.*, **44**, 95–104.

Goldstein Goldstein Dreher

GOSSELINK, R.J.A., VAN DAM, J.E.G., DE JONG, E., GELLERSTEDT, G., SCOTT, E.L., SANDERS, J.P.M. (2011) Effect of periodate on lignin for wood adhesive application. *Holzforschung*, **65**, 155–162.

HADI, Y., WESTIN, M., RASIYD, E. (2005) Resistance of furfurylated wood to termite attack. *Forest Prod. J.*, **55**, 85–88.

INOUE, M., NORIMOTO, M., TANAHASHI, M., ROWELL, R.M. (1993a) Steam or heat fixation of compressed wood. *Wood Fiber Sci.*, **25**, 224–235.

INOUE, M., OGATA, S., KAWAI, S., ROWELL, R.M., NORIMOTO, M. (1993b) Fixation of compressed wood using melamin formaldehyde resin. *Wood Fiber Sci.*, **25**, 404–410.

INOUE, M., SEKINO, N., MOROOKA, T., ROWELL, R.M., NORIMOTO, M. (2008) Fixation of compressive deformation in wood by presteaming. *J. Trop. For. Sci.*, **20**, 273–281.

KAMKE, F.A. (2006) Densified radiata pine for structural composites. *Maderas-Cienc. Technol.*, **8**, 83–92.

KIM, M.G., BOYD, G., STRICKLAND, R. (1994) Adhesive properties of furfural-modified phenol-formaldehyde resins as oriented strandboard binders. *Holzforschung*, **48**, 262–267.

LANDE, S., WESTIN, M., SCHNEIDER, M. (2004) Properties of furfurylated wood. *Scand. J. Forest. Res.*, **19**, 22–30.

NAVI, P., GIRARDET, F. (2000) Effects of thermo-hydro-mechanical treatment on the structure and properties of wood. *Holzforschung*, **54**, 287–293.

SCHNEIDER, M. H. 1995. New cell wall and cell lumen wood polymer composites. *J. Wood Sci. Technol.*, **29**, 135-158.

SEBORG, R., MILLET, M., STAMM, A. (1945) Heat stabilized compressed wood – Staypak. *Mech. Eng.*, **67**, 25–31.

SHAMS, M.I., YANO, H., ENDOU, K. (2004) Compressive deformation of wood impregnated with low molecular weight phenol formaldehyde (PF) resin I: effects of pressing pressure and pressure holding. *J. Wood Sci.*, **50**, 337–342

STAMM, A.J., SEBORG, R.M. (1939) Resin-treated plywood. Ind. Eng. Chem., **31**, 897–902.

VENAS, T.M., FELBY, C. (2009) Enzymatic Hydrolysis of Furfurylated Scots Pine Sapwood (Pinus sylvestris, L.). In: *Proceedings of the 4th European Conference on Wood Modification*. Stockholm, Sweden.

VORREITER, L. (1934) Lignostone, das neue Holzveredlungserzeugnis. *Forstw. Cbl.*, **56**, 533–542.

WELZBACHER, C., WEHSENER, J., RAPP, A., HALLER, P. (2008) Thermomechanical densification combined with thermal modification of Norway spruce (Picea abies Karst) in industrial scale – Dimensional stability and durability aspects. *Holz. Roh. Werkst.*, **66**, 39–49.

WESTIN, M., OHLSSON, B., SIMONSON, R. & NILSSON, T. (1996). New chemicals for wood preservation and new ways of chemical modification. In: *Proceedings of the 212th ACS National Meeting*, Orlando, USA, **Part I**, Cell: 006.

# Increasing the value of hardwood veneers by heating treatment\*

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### ABSTRACT

At the manufacture and use of veneers an essential aspects the pattern, color and homogeneity of the wood. These qualities influence the value of a veneer.

The simplest way to change the colour of the wood is a modifier procedure. Thanks to the thickness of veneers treating is much faster and easier than a larger wood. Dry heat treatment was applied during the research taking into account of industrial applications.

The project aims is to determine the color change depending on treatment temperature and time. The research work covered the Hungary most widely used veneer (oak, ash, beech, cherry and maple). Four different temperatures (80, 120, 160, 200°C) with 12 period (5, 10, 15 ... 60 minutes) were applied. The color change is measured by CIELAB color stimulus measuring system.

Our results showed that the used treatment time and temperature together determine the final coloration. Higher temperatures need shorter periods of heat treatment to achieve the same color as a lower temperature, but prolonged treatment. At lower temperatures one-hour treatment period causes only a color change barely noticeable for the human eye. At higher temperatures the change of the color stimulus is much more spectacular and is closely related to the treatment time (0.9 min).

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Different tree species respond differently to treatment. At different temperatures the measured color change resulted different order of the tree species.

### INTRODUCTION

The color of objects has extremely important role in everydays life. The color is often achieved using the appropriate paints.

The wood gentled with heat treatment is flourishing today. Across Europe, increasing the demand for heat-treated wood materials which have dark color, like tropical woods. In this study our aims were to define treatment parameters which cause noticeable color change at short period for veneers.

The heat treatment of wood is heat-driven degradation of it, while the parameters are under control and where the internal and external factors have relevant importance. As the medium of the treatment vegetable oil (MENZ Holz AG-Germany), normal FWD procedure (pl. PLATO®-Netherlands), dry treatments with steam vaporing (Thermo Wood®-Finnland) or treatment in nitrogen (Retified Wood®-France) are used.

In the industry generally 230°C is marked as the top of operating temperature. The processes above 200°C have minimal beneficial effect on the profitability and the properties of the final products. Below 200°C carbon dioxide raises in small quantities, and with the increasing of the temperature combustible gases, such as carbon monoxide and methane, are also formed during the decomposition. There is no significant weight loss and gas formation in the range of the process of moderate temperature (100-200°C) although some of the characteristics of the wood, mainly the color change significantly (NÉMETH 1998). This can be observed both wet and dry wood, but the color change in case of of dry wood was lower (TOLVAJ ET AL. 2010).

The wood discoloration caused by heat between 100 and 200°C mainly due to chemical transformation of the additional substantials. The color change between 160 and 180°C increases rapidly both in inert (NÉMETH ET AL. 2009) and oxidative atmosphere. It caused by the decompositions of the additional substantials and supplemented with the effects of leaching. The steaming of most species also results homogeneity (for example the black locust and cherry) (DIANISKOVA ET AL. 2008). The color characteristics are varying depending on the applied atmosphere and the wood species as follows: the light of the wood significantly decreases; the coloration of the rate of change significantly reduces and the color of the wood converge to the limit, specified by the species and treatment time. A really long treatment

period gives similar color-stimulus properties as the higher temperature (NÉMETH 1998).

KOLLMANN ET AL. (1951) has detailed information about the thermal treatment induced color change of wood first time. The rate and extent of

discoloration is determined by of the applied temperature, because at higher temperatures the thermal processes accelerate, so the color change will be more intense. They found that the color change increases exponentially by the temperature increase.

BOURGOIS ET AL. found that the decrease of the brightness and the color shift at heat treatment (240-310°C) mainly caused the decreasing amount of the hemicellulose and especially the pentose (CSONKÁNÉ 2005).

NÉMETH established in his studies (1989a, 1989b, 1998) that during the thermal treatment the change the brightness of the wood gives the most information and this value is the closest to the subjective color awarding data. CSONKÁNÉ (2005) examined the high extract-contenting black locust and lower extract-containing black poplar species and shown that the color changing of the no extract-contenting black locust similar to the poplar's change.

A similar analysis of quercetin and robinetin found that the hardwoods can be classified into two groups by the extract compounds which changes similar to these two model compounds. The color change of poplar wood illustrates quercetin-type changes because this species contains only small amounts of extract compound, so the color change can thanks to practically the lignin and to extract the contents (containing only slight amount of chromophore group). The extracted black locust speciments also showed quercetin-type color change. In the extract-free wood (because the absence of color-forming compounds) fast starts the decomposing of the hemicellulose and lignin and the relationships of them, which leads to the formation of chromophores. This the explanation of the slow, but continuous color-change of this type of wood. The black locust wood shows robinetintype change, which has a great amount of, mostly colored, extract compounds (CSONKÁNÉ 2005).

NIEMZ (2004) treated spruce specimens in different media and found that they have different colors from the 200°C treatments. The resulting samples then were undergone outdoor tests, where the heat-treated specimens have lower cracking tendency, but both the untreated and the treated specimens became similarly gray. NIEMZ said that the color of the modified wood is not UV stable.

# **EXPERIMENTAL METHODS**

The visual perception of the color largely depends on the biological parameters of the percipient. An objective method is assessing the colors on systems. Among the objective color determination the most modern methods is the instrumental color measurement, where the color parameters can be expressed in numbers.

The research work tested the most popular veneer materials (oak, ash, beech, cherry and maple) in Hungary. On successive – by shedding from blade - veneer samples ( $4 \times 48$  db,  $500 \times 100 \times 1$  mm), the color were measured on the same side and on 10-10 template pre-designated place, with Konica Minolta's CM-2600-type device which uses the CIELAB color-measurement system. The heat treatments occurred in Memmert UFP-400 device. Measurements were done on 80-120-160-200°C in 12 treatment period (in 5 minutes).

We specified the rate of the changing a color by the difference of the values in of CIELAB system ( $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ). We also specified the cumulative effect of differences by calculating the value of stimulus color difference ( $\Delta E^*$ )

# **RESULT AND DISCUSSION**

There are not seen significant changes in the color coordinates at treatments between 80 and 120°C. The resulted differences in color coordinate clearly defined by the differences in the baseline, control color. This is observed in the case of all color of the components regardless of species. A further trend in color change is predicted on 120°C at the last measurement points (50-55-60 min) the observed increasing changes in color coordinates of some species. It should be noted that on these low-temperature treatment only decrease of the oak's, L\* (lightness factor) is striking, but the other two color factor aren't change.

The spectacular change of the components of color-stimulus is clearly observed at 160°C. For each of the studied species can be said that with the  $a^*$ ,  $b^*$  coordinates change in a positive direction – in proportion of increasing the treatment time – the color will increasingly shift towards the red and yellow, with the decreasing of L\* (wood is darkening).

In the a\*, namely in green-red color change, the cherry and maple have prominent change aoung the studied species. The rate of change of the color coordinates gradually slows down from the initial intensity after ca. 30 minutes. The a\* color coordinate (red content) increased to 2-3 times higher in case of oak, beech and ash veneer, and 5-6 times higher in case of cherry and maple (60 min treatment). At 200°C in all treatment time also the maple's a\* component shows the greatest change (tenfold), while in case of cherry increased very little compared to the measured values at 160°C. The change of the red color component of ash samples accelerate over the time of treatment and become similar to the cherry (6-7 fold). The change rate of a\* of beech and oak specimens is similar to the modification in 160°C.

In case of the b\* (blue-yellow) axis of the spatial coordinate system of color maple shows the greatest value at 160°C, the change is 3-4 times greater compared to the other studied species already at the first 5-10 minutes of the treatment period. This difference remains until the end of the maximum treatment period. The yellow color change of beech, ash, cherry and oak veneers were on the same (from 2 to 2.5-fold) level.

At the highest temperature, the change of  $b^*$  of the maple speciments reaches 5-6 fold value at the shortest (5 min) time interval, but substantial and constant increase begins only 30-35 minutes after the start of heat. The final value is from 11 to 12 times of the baseline. The yellow color component of the ash samples continuously grow – as at 160°C - but the rate of it almost twice. The values of other investigated species have not changed or are not significantly decreased.

There were no significant differences in the change of the lightness factor  $(L^*)$  of the different species at the same temperature and treatment time. The decreasing rate of the value of  $L^*$  is continuous (at 160°C, after 30 min treatment) in the case of cherry, while at the other species it slows down with the time. This continuity is observed at each species (Fig. 1), at 200°C, the decrease of rate occurs only by the beech.



Figure 1: Color change of oak veneer with treatment time

The difference of color-stimulus ( $\Delta E^*$ ) shows the combined effects of color change. At 80°C the calculated values do not show a clear relation to the treatment period. The differences between the measured values reflect to the differences between the control samples regardless of species. A closer relationship has observed at 120°C heat treatment, in case of the oak and beech, but the differences between control samples have significant effect. The observation that the color change between 160 and 180°C increases by leaps and bounds (NÉMETH 1998) is correct for the veneers too, as at 160°C heat-treated samples the measured differences grows significantly and significant the differences between the species.

There was a close relationship between the measured values in time (Table 1).

	Beech	Cherry	Ash	Oak	Maple
160°C	0,9805	0,9901	0,9603	0,8541	0,9824
200°C	0,9383	0,9363	0,8446	0,9148	0,9056

Compared to the initial state at 160°C until 30-35 minutes the intensity of the color change is similar at all investigated species. After 1 hour treatment the cherry and the maple showed the greatest change but the cherry's rate of changing became more intense at the end of the treatment. The ash has the lowest change despite it's bright hue (Fig. 2).

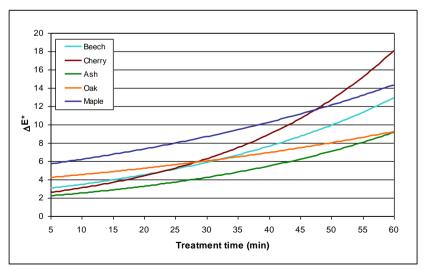


Figure 2: Total color change at 160°C

At 200°C heat treatment – similarly to the 160°C - the color change become more intense after 30-35 minutes, but the oak and ash has the highest rate. Changes in the beech is the most balanced, but the final value is significantly lower than other species' (Fig. 3).

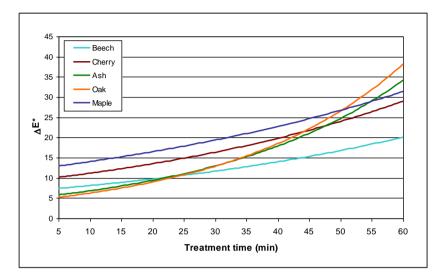


Figure 3: Total color change at 200°C

Seeing the change of color components at oak, cherry and maple at 160 and 200°C heat treatment you can see that these components determine the  $\Delta E^*$  in different percentage in the different species. Of course the change changes over the time and the heat treatment temperature also (Fig. 4-5).

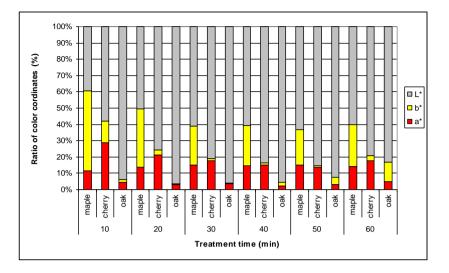


Figure 4: The ratio of color coordinates determining  $\Delta E^*$  at 160°C

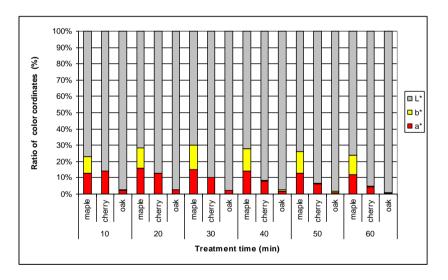


Figure 5: The ratio of color coordinates determining  $\Delta E^*$  at 200°C

Until at 160°C the the color stimulus differences is significantly affected by yellow and red hues change, while at higher temperature it will primarily resulted by changes in light. At both treatment temperature  $a^*$  and  $b^*$  is decreasing, while the L\* coordinate increasing with time. These changes differ in different species. In case of the maple at 160°C after 1

hour - steadily declining rate of participation - the yellow and red hues of the color stimuli give the 40% of the difference. In case of the cherry and oak, this is only 20%. At the higher heat treatment in case of the maple seems primarily the role of  $a^*$  and  $b^*$  compared to the L \*, but the importance has declined. In case of the oak and cherry at higher temperature the yellow hue changes can completely neglect.

### CONCLUSION

The heat treatment of the veneer quickly and easily enforced because of the geometrical dimensions because it can easily reach the medium temperature in a relatively short period and over the entire cross-section. So the change (hemicellulose, cellulose, lignin, etc.) can be done over the entire cross-section soon. This easily feasible technology is able to the increase the worth of the lower-value wood products.

We were looking for the lower limit of the low temperature modifications which has a visible color differences because in industrial applications the required energy and the treatment period is a significant factor. Not negligible that these modifying treatments for aesthetic color can help to replace deep-toned exotic species with domestic hardwoods. It can be costeffective and can stimulate the domestic economy.

We demonstrated by our measurements that below 120°C only for the human eye can barely noticeable color change can be reach with 1 hour period. This change can be seen in the case of the oak and maple samples, barely detectable in other species.

At 160 and 200°C, in general, the color-stimulus difference is visible (3 <) or large (6 <) as the treatment period increases. The two treatment temperature caused the most intense change by different species. The oak has the lowest color change at 160°C, but at higher temperature has the biggest. For this two temperature, in case of all species the change of color-stimulus have relation to the treatment period (correlation coefficient higher than 0.9).

In it plays a large role that we were measured the color coordinates uniformly the outer side (cylindrical) of the veneers. In fact, some studies have shown (THOMPSON et al. 2005) that rate of the changes are affected by the place and the anatomical direction of the raw material in the timber.

### REFERENCES

BOURGOIS, P.J., JANIN, G., GUYONNET, R. (1991): La Mesure de Couleur. Une Méthode d'Etude et d'Optimisation des Transformations Chimiques du Bois Thermolysé. Holzforschung 45., 377-382.

CSONKANE, R.R. (2005): A flavonok és a faanyag termikus átalakulása. PhD értekezés, NYME, Sopron.

DIANISKOVA, M., BABIAK, M., TOLVAJ L., (2008): Color homogenisation of cherrywood (Cerasus avium L.) and black locust (Robinia pseudoacacia L.) during steaming. Wood Research Volume 53(4), 45-58.

KOLLMANN, F., KEYLWERTH, R., KÜBLER, H. (1951): Verfarbungg des Vollholzes und der Furniere bei der Künstlichen Holztrocknung. Holz als Roh- und Werkstoff 9., 382-391.

NÉMETH, K. (1989A): A faanyag abiotikus degradációja. Doktori Értekezés, Sopron

NÉMETH, K. (1989B): A faanyagok fotodegradációja. Faipar 39(11), 330-332.

NÉMETH, K. (1998): A faanyag degradációja. Mezőgazdasági Szaktudás Kiadó. Budapest.

NÉMETH, R., BAK, M., TOLVAJ, L., MOLNÁR, S. (2009): The effect of thermal treatment using vegetable oils on physical and mechanical properties of Poplar and Robinia wood. ProLigno Volume 5(2), 33-37.

NIEMTZ, P. (2004): Vortrëge an "Thermoholz Workshop" 2004-2006, Tagungsbände – CD, IHD Dresden.

THOMPSON, W.D., KOZAK, A.R., EVANS, D.P. (2005): Thermal Modification of Color in Red Alder Veneer. I. Effects of Temperature, Heating Time, and Wood Type. Wood and Fiber Science, 37, 53-661.

TOLVAJ, L., MOLNÁR, S., NÉMETH, R., VARGA, D. (2010): Color modification of black locust depending on the steaming parameters. Wood Research 55(2), 81-88.

# Wood modification at the University of West Hungary\*

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**Keywords:** wood modification, heat treatment, acetylation, THM treatment, nanozinc

# ABSTRACT

Wood modification in different mediums dates back from decades at the University of West Hungary (Faculty of Wood Sciences, Institute of Wood Science). First investigations were high pressure and temperature steaming, and ammonia treatment of black locust. The demand for modified wood material increased Europe-wide, due to the commercialisation of different wood modification processes in the last decades. We have made complex investigations in terms of Hungarian hardwoods' heat treatment in the project "Wood preservation without chemicals" (supported by GVOP). Due to this project, also industrial developments were established in 2007 in Hungary. The aim of the industrial development was to elaborate heat treatment schedules for black locust and to build a prototype of heat treatment equipment. Wood modification indicates a lot of research objective in our institute. Wood modification processes indicate continuously new challenges. In the last years we gave special attention to heat treatment processes in vegetable oils and paraffin, acetylation and some impregnation processes. As in the last years nanotechnology came to the front, we investigated the application possibility of nano-scale materials in wood industry.

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# INTRODUCTION

Wood is recognised as the most important of the renewable base materials with the added advantage of being recyclable and CO2-neutral. It is a highly versatile material and as such has been utilized in building and construction. The demand for timber is continually increasing, especially in slower growing hardwood and tropical species. Such species offer a greater durability and higher aesthetic qualities than many of the faster growing softwood species. It is well known that there are grave ecological and environmental concerns over current 'virgin timber' demands, and various attempts are underway to prevent the demise of many of the biologically diverse regions where these timbers originate. A greater emphasis is now being placed in sustainable harvesting of timber species, though the slow growth of many species means a slow turnover in materials and profits. Thus it is necessary to encourage the use of faster growing timbers which may be readily gained from such sustainable plantations. But wood is a biodegradable material. Degradation leads to the reduction in strength and the increased risk in structural decay through fungal infestation. Many traditional protection treatments currently exist to prevent these deteriorations, but often they are based on toxic materials. Apart from the risks involved in using such materials for treatments, there is increasing concern over the problems arising in the disposal of the timbers after the end of their commercial lifetime. Whilst the timber sector recognizes these increasing safety requirements, it also realizes a lot of research and development will be required in overcoming these problems. Wood modification is a generic term describing the application of chemical, physical, or biological methods to alter the properties of the material. The aim is to get better performance from the wood, resulting in improvements in dimensional stability, decay resistance, weathering resistance, etc. It is essential that the modified wood is non-toxic in service and the disposal at the end of life does not result in the generation of any toxic residues (HILL 2011).

# INVESTIGATED MODIFICATION PROCESSES

### Heat treatment in gaseous atmospheres

The institute possesses a programmable heat treatment chamber (Figure 1.) in which it is possible the treatment of maximum 60cm long samples. In this electric heated, supplied with ventilators and control panels equipment it is executable a heat treatment in normal atmospherical air. Due to the

elaborated schedules and 6 years experience the good quality of heat treated wood is secured.



Figure 1: Experimental heat treatment chamber

In 2010 a combined heat treatment-steaming equipment with 0,5m<sup>3</sup> capacity were purchased (Figure 2). This autoclave is suitable for heat treatments up to 250°C temperature in vacuum, inert gases and steam. Investigated wood species so far: oak, turkey oak, black locust, poplar, hornbeam, beech, maple, pine and spruce. As a result of the treatments durability was improved remarkably (Figure 3.) and swelling decreased as well.



Figure 2: Combined treatment autoclave

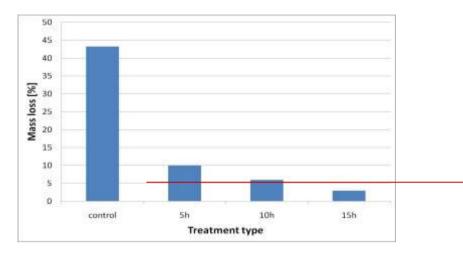


Figure 3: Improvement in durability of poplar wood against Coriolus versicolor (treatment temperature: 200°C) (Horváth et al. 2006)

By means of heat treatments exotic and homogeneous colour can be achieved in whole cross-section of the wood. This property was very useful by producing of flooring elements from the material heat treated in our institute (Figure 4). Beside of the favourable properties the bending, tensile (20-40%) and impact bending strength (30-70%) was decreased considerably. However, hardness and compressing strength increased slightly.



Figure 4: Flooring elements with heat treated oak, turkey oak, beech and ash top-layer (left to right)

### Heat treatment in different fluids

Efficiency of heat treatment processes depends on the rate and regularity of the heat growth in the wood, and on the reducing of oxidative processes in the interest of avoid unreasonable decomposition. Heat treatment in vegetable oils can be a solution for these problems. Wood was heat treated in rapeseed-, linseed- and sunflower oil at 160-200°C. Swelling properties decreased 20-60% and strength decreased less than by heat treatment in a gaseous atmosphere. Colour changes were similar than by heat treatment in vegetable oils is the short treatment time (by a 25mm thick poplar board, up to 6 hours including drying). However, it have to be noted, that for example by black locust, which has non-porous structure, longer treatment times are needed to avoid cracks and deformations (Bak et al. 2009). With applying paraffin as heat treatment medium instead of vegetable oils, similar results can be achieved as well as moisture uptake decreased further because of the thin paraffin layer on the surface (Németh et al. 2012).



Figure 5: Colur change of poplar wood due to different heat treatments in linseed oil

# Thermo-Higro-Mechanical (THM) treatment of wood

In terms of each product often only one property is important to be suitable for the requirements. In terms of poplar woods indoor use surface hardness is the property which limits the utilization. The goal in this case was to produce a material with low density and high surface hardness. With Thermo-Higro-Mechanical treatment – using heat, steam and compressing on wood – hardness of poplar wood can be increased from the very low 10 N/mm<sup>2</sup> to 22 N/mm<sup>2</sup>. With 30% compression of poplar wood hardness increases 120% and reaches hardness of maple wood, which is a popular wood species of flooring element construction. Beside of the improved surface hardness wood colour became brown in 2-3mm deep (Ábrahám et al. 2010 Edinburgh).

### Acetylation of wood

One of the most common chemical modification processes is acetylation, which changes –OH groups in wood to acetil-groups. In the centre of our investigations were black locust and poplar. By poplar wood decreased swelling 70%, beside that the mechanical properties remained unchanged. The black locust cannot effectively treat as solid wood because of the small penetration deepness due to its compact structure. However, as veneer or flake good result can be achieved (eg. producing of weather resistant plates). The process demands special equipment and its complicated (Figure 6.), furthermore for purchasing of acetic-anhidride authorization is required. Treated material typically lose colour, but with appropriate surface finishing can be brighted up. Wood has acetic smell for a long time (evaporation), furthermore by application of hinges increased corrosion have to be calculated.



Figure 6: Reactor for acetylation

### Other wood modification processes

In addition to the modification processes above, we have experience in some other processes to. First of all, impregnation processes with beeswax (Figure 7) or nanozinc-particles can be highlighted. Treatment with nanozinc particles were developed together with the Institute of Wood and Paper Technology in Sopron. Both treatments has the goal to improve fungal resistance of wood.



Figure 7: Impregnation with beeswax

Zinc-nanoparticles improved durability very effective because already very low concentrations (0,22 and 0,055m/m%) resulted in significant resistance against decay (Bak et al. 2012) (Figure 8). Investigation of treatment with beeswax in terms of durability is currently in process, however it can be mentioned as a positive result that the process decreases moisture uptake of wood significantly (10-40%). If fungal decay were preventable with beeswax impregnation, it were a natural based preservative for wood without any chemicals.



Figure 8: Anti-fungal effect of nanozinc (left: treated; right: untreated)

	Durability	Density	Hardness (butt)	Bending strength	Swelling [%]		
	[EN 350-2]	[kg/m <sup>3</sup> ]	[N/mm <sup>2</sup> ]	[N/mm <sup>2</sup> ]	radial	tangential	
Teak	1	520-660-700	63-71	58-109	2,1-3,0	4,2-5,8	
Pine	3-4	330-510-890	35-40-95	41-80-205	3,3-4,5	7,5-8,7	
Robinia	1-2	580-770-900	67-78-88	103-136-169	3,2-4,6	5,4-7,2	
Beech	5	540-720-910	~72	74-123-210	~5,8	~11,8	
Poplar	5	~410	20-25	60-75	3,3-4,7-5,8	4,9-6,7-8,9	
THM poplar (20%)	-	~490	22 (side)	84-119	-	-	
OHT poplar	2-3	~380-480	-	40-95	2,1-3,1-4,2	4,3-4,9-5,7	
OHT robinia	-	~670-760	-	60-150	3,3-4,1-5,7	4,2-5,1-6,4	
Thermowood beech	1-2	~720-771	-	100-135	3,6-5,1	7,2-11,8	
Acetylated pine	1	-	-	80	0,4	0,7	
Acetilated beech	1	-	-	115	0,7	1,1	

Table 1:	Some pro	perties of	f natural	and treated	l timber
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### CONCLUSIONS

At the University of West Hungary (Institute of Wood Science) important research activity were executed in the last 10 years in terms of wood modification. In course of that, effects of numerous modification processes were investigated on wood. The main topic were investigation of different heat treatments (heat treatment in different gaseous atmospheres or liquids and with compressing), but we achieved also some good results in terms of acetylation and in development of environmental friendly wood preservatives (beeswax, nano-zinc particles) too. Some properties of treated wood are shown in Table 1...

#### REFERENCES

ÁBRAHÁM, J.; NÉMETH, R.; MOLNÁR, S. (2010) Thermo-mechanical densification of Pannónia Poplar. In: Proceedings of the final conference of COST Action E53: 'Quality control for wood & wood products' 4-7th May 2010., Edinburgh. pp. 282-292.

BAK, M.; YIMMOU, B.M.; CSUPOR, K.; NÉMETH, R.; CSÓKA, L. (2012) Enhancing the durability of wood against wood destroying fungi using nanozink. In: International Scientific Conference on Sustainable Development & Ecological Footprint. Sopron, Hungary, 26-27.03.2012.

HORVÁTH, N.; CSUPOR, K.; MOLNÁR, S.; NÉMETH, R; (2006) The effect of thermal modification on the durability of wood against fungal decay. *Proceedings of the 5th International Symposium Wood Structure and Properties '06.* Technical University in Zvolen, Slovakia

NÉMETH, R.; BAK, M.; TOLVAJ, L.; MOLNÁR, S. (2009) The effect of thermal treatment using vegetable oils on physical and mechanical properties of Poplar and Robinia wood. *Pro Ligno*, **5**(2), 33-37.

NÉMETH, R.; BAK, M.; CSORDÓS, D. (2012) Thermische Modifizierung von Buche und Pappel mittels Paraffin. *Holztechnologie*, **53**(under press)

# Diagnostic features of European beech with wavygrained wood growing in Ukrainian Carpathian

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**Keywords:** beech, wave-grained wood, decorative wood, forest stands, diagnostic model

#### ABSTRACT

The wave grain abnormality in European beech (*Fagus sylvatica* L.) can greatly enhance its commercial appeal. However, beech with wavygrained wood has been as pulpwood exploited, without exploring management strategies that can improve its potential. Even though the initiation and development processes of wavy-grained wood of beech are still largely unknown, useful silvicultural advice can still be provided for forest managers. The identification of beech with wavy-grained wood before felling, or early in the merchandising process, allows for better protection of log value. Wavy-grained wood abundance could be associated with forest management; it may prove advantageous to keep known curly beech to ensure continued decorative wood production.

#### **INTRODUCTION**

*Fagus sylvatica* L. is one of the main hardwood trees species in Ukraine growing in 525 thousand ha. There are about 32 beech forest types (HERUSHYNSKYY 1996, OSTAPENKO ET AL. 1998). The natural beech forest are mostly concentrated in Carpathian mountains from 250 to about 1250 m about see level. In scientific point of view it is a unique natural lab to carry out the wood quality in the wide meaning. There are many research project of European beech in subject area forestry, silviculture, wood properties and quality, but only a few of them about beech with wave-grained wood (COST E 53 2007, COST E42 2008). Wavy-grained wood is characterized by the unique color, grain and texture that give the appearance of undulating waves as they reflect light differently (Fig. 1). The abnormality of wood significantly increases its commercial appeal (KOHL 2009, SOPUSHYSNKYY 2012).



Figure 1: European beech with waved-grain wood (a) growing trees and (b) wood texture

In Ukraine decorative wood is currently undervalued and has been as pulpwood exploited, without exploring management strategies that can improve its potential. Knowing the special features of decorative wood foresters can manage stands with curly beech linking to the target consumers. Hence, the economic value of decorative wood should be corresponded to its real market price. Even though the initiation and development processes of wavy-grained wood of beech are still largely unknown, useful silvicultural advice can still be provided for forest managers. The identification of beech with wavygrained wood before felling, or early in the merchandising process, allows for better protection of log value. Wavy-grained wood abundance could be associated with forest management; it may prove advantageous to keep known curly beech to ensure continued decorative wood production. Additionally, by discerning high-value decorative characteristics one could gain significant competitive advantage. For example, furniture, cabinet, moulding and millwork and other value-added manufacturing industries in Europe can take the opportunity to make advantage of the attribute findings. Increased utilization of the locally grown beech trees may result in profitability for the forestry and wood processing enterprises.

Biological and physical characteristics of beech wavy-grained wood and its distribution have not been subject of special study, and scientific and practical recommendations for its diagnosis, care, conservation and forestry can provide great insight to the forest managers. The identification of this type of wood early in the forestry process allows better log value protection. There is a lack of peer-reviewed, scientific investigations of the silviculture of beech stands with wavy-grained wood.

The main objectives of this study are to develop diagnostic features for beech wavy-grained wood and to substantiate advices for the management related to the growing curly beech in the Ukrainian Carpathian forest.

#### MATERIALS AND METHODS

The study was conducted in the natural beech stands growing in the Ukrainian Carpathian (48°18′20″N 25°25′09″E, 400 m above sea level). Silvicultural and dendrometrical characteristics of broadleaves forest stands were collected by field studies with focus on acquiring and in-depth understanding of beech with wavy-grained wood as well as its forestry potential. Altogether, 6 model trees of beech with wavy-grained wood and 6 of beech straight-grained wood were studied for their morphological features. To systematize knowledge about differences in morphological characteristics of curly beech has been used some specific features (Fig. 2).

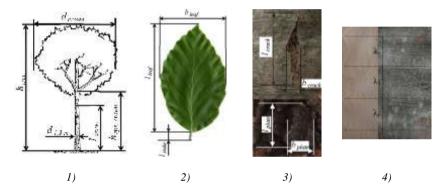


Fig. 2. Specific biometric features of European beech: 1) tree  $(l_{an.w.} - length of wood anomaly, m; h_{tree} - height of tree, m; h_{spr. crown} - spring of crown, m; d_{crown} - average diameter of crown, m; d_{1,3m} - diameter at the breast height, cm); 2) leaf <math>(l_{leaf} - length of leaf, cm; l_{stake} - length of leaf stake, cm; b_{leaf} - width of leaf, cm; 3) bark (l_{crack} - length of bark crack, mm; b_{crack} - idth of crack, mm; l_{plate} - length of bark plate along the axle-tree, mm; b_{plate} - width of bark plate transverse to the tree, mm; 4) curly grain (<math>\lambda_i$  - length of wave, mm).

The bark thickness  $(t_{bark}, mm)$  was determined in the beech trees with smooth and fissured bark. Leafs were collected at the bottom, center and top of the crown. Three variables as the ratio between:  $l_{leaf} / b_{leaf}$ ,  $l_{leaf} / l_{stake}$  and  $b_{leaf} / l_{stake}$  were determined. The morphological characteristics of leaf, wood and bark were measured by software AutoCAD 8.0 on the digital photos.

#### **RESULTS AND DISCUSSION**

Morphological characteristic of trees are the best diagnostic features of wood quality. They are mainly influenced by genetic and environmental factors. The genetic of hereditary variation of beech is well characterized by straight-grained and wavy-grained wood and reflected by morphological distinctions (Table 1). The wavy-grained wood of European beech is characterized by valuable decorative features.

Variables	Wood	N, units	min	$M^{\pm m}$	max	V, %	P, %
Tree							
l <sub>an.w.</sub> [m]	wavy-grained	18	5,0	$5,9^{\pm0,21}$	8,0	15,4	3,6
h [m]	straight-grained	18	22,0	25,9 <sup>±0,40</sup>	28,0	6,5	1,5
h <sub>tree</sub> [m]	wavy-grained	18	17,5	$20,7^{\pm0,36}$	23,0	7,4	1,7
4 [aur]	straight-grained	18	26,0	$31,4^{\pm0,59}$	36,0	8,0	1,9
$d_{1,3m}$ [cm]	wavy-grained	18	29,0	$37,1^{\pm0,92}$	43,0	10,5	2,5
h []	straight-grained	18	9,0	$12,8^{\pm0,39}$	16,0	13,0	3,1
h <sub>spr. crown</sub> [m]	wavy-grained	18	5,0	7,1 <sup>±0,23</sup>	9,0	13,7	3,2
J []	straight-grained	18	7,0	$8.0^{\pm0,16}$	9,5	8,7	2,1
d <sub>crown</sub> [m]	wavy-grained	18	6,5	$7,6^{\pm0,19}$	9,0	10,5	2,5
	·	Woo	od			•	
$\lambda$ [mm]	wavy-grained	351	25,4	$58,0^{\pm0,98}$	112,4	31,7	1,7
		Bai	rk				
t [mm]	straight-grained	77	8,0	$12,4^{\pm 0,31}$	21,2	22,0	2,5
t <sub>bark</sub> [mm]	wavy-grained	75	4,6	$7,8^{\pm0,15}$	12,1	17,1	2,0
l <sub>crack</sub> [mm]	wavy-grained	100	78,9	$164,8^{\pm4,78}$	282,8	29,0	2,9
b <sub>crack</sub> [mm]	wavy-grained	100	4,3	31,0 <sup>±1,42</sup>	71,5	45,7	4,6
L <sub>plate</sub> [mm]	wavy-grained	47	15,7	$22,4^{\pm0,74}$	48,2	33,2	3,3
b <sub>plate</sub> [mm]	nm] wavy-grained		7,9	13,9 <sup>±0,16</sup>	20,2	21,3	1,1
Leaf							
l <sub>leaf</sub> / b <sub>leaf</sub>	straight-grained	289	1,14	$1,53^{\pm 0,01}$	2,24	9,1	0,5
Ileaf / Dleaf	wavy-grained	406	1,25	$1,62^{\pm 0,01}$	2,33	8,1	0,4
1/1 .	straight-grained	289	4,06	$7,51^{\pm0,09}$	11,75	20,7	1,2
l <sub>leaf</sub> / l <sub>.stake</sub>	wavy-grained	406	3,87	$6,99^{\pm0,08}$	10,87	23,5	1,2
bleaf / lstake	straight-grained	289	2,78	$4,96^{\pm0,07}$	7,73	22,9	1,3
Uleaf / Istake	wavy-grained	406	2,18	$4,34^{\pm 0,05}$	6,73	25,2	1,3

 Table 1: Morphological distinctions of wavy- and straight-grained beech wood

Note: N - Number of samples; min – minimum; M±m - mean and its standard deviation; max – maximum; V – coefficient of variation; P – accuracy figure.

The beech trees with wavy-grained wood are characterized by smaller height and bigger diameter at the breast height on 20 and 18 % respectively. The spring of crown of curly beech started at the average height of 7,1 m and the length of anomaly wood is in range of 5-7 m. There are no significant differences in the diameter of crown.

The ration  $l_{leaf} / b_{leaf}$  is more on 6 % in the curly beech, and  $l_{leaf} / l_{stake}$  and  $b_{leaf} / l_{stake} - less$  on 7 and 13 % respectively. The average length of wave is

estimated about 58 mm. The curly beech is characterized by fissured bark and the ratio of length and width is about 5.

# CONCLUSIONS.

European beech with wavy-grained wood is valuable decorative material for veneer production. The morphological characteristics of beech trees with wavy-grained and straight-grained wood are significant differentiated. The identification and production of high decorative timber is to be one of the economic and strategic lines for the sustainable forestry and wood processing.

# REFERENCES

COST E42. (2008) *Growing valuable broadleaved tree species* [Final Report] / Edited by G. Hemery, H. Spiecker, E. Aldinger [et al.]. - Freiburg im Br.: Uni-Freiburg, 2008. – 40 p.

HERUSHYNSKYY Z.YU. (1996) *Forest typology of Ukrainian Carpathians*. Lviv, Piramyda, 208 p.

KOHL F. (2009) Furnier – Tradition mir Netzwerk und Perspektiven. *Furnier Magazin*, 32-49.

OSTAPENKO B.F., FEDETS I.P., PASTERNAK V.P. (1998) *Typology diversity of Ukrainian forest. Зона широколистяних лісів.* Kharkiv, Agricultural University, 127 p.

COST E 53. (2007) *Quality Control for Wood and Wood Products*: The first conference COST E 53, 15th -17th October 2007, Warsaw / Edited by M. Grześkiewicz. – Warsaw University of Life Sciences, Faculty of Wood Technology, 2007. – 173 p.

SOPUSHYSNKYY I.M. (2012) Grading and assessment of decorative wood quality: sycamore (Acer pseudoplatanus L.), beech (Fagus sylvatica L.) and ash (Fraxinus excelsior L.). Scientific magazine, Scientific technical collection, 22.2, Lviv, UNFU, 106-111.

# Quality assessment of beech logs using CT-scanning technology

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Keywords: beech; timber quality; CT; knottiness; accuracy

# ABSTRACT

As part of the "Flexwood" project the use of X-ray computed tomography (CT) for log quality assessment was tested as one stage within the forestwood supply chain to provide information on raw material prior to processing. The objective of the project was to match information at different stages in the supply chain to fulfil customer demands.

The approach aims at developing a system which can provide reliable information on beech timber quality, such as knottiness, prior to primary breakdown sawing. For this purpose relationships between the external appearance of logs and log internal features were established.

After felling trees, branch scars were manually assessed on the round wood prior to CT scanning the logs. The images of these scans were analysed to identify position and size of knots. These CT-derived knot characteristics were checked for accuracy through validation measurements on sawn timber and radial cuts.

The validation shows a high level of accuracy in quantitative and qualitative knot assessment using CT-scanning technology. The measurements additionally confirmed that external branch scar characteristics are closely related to the proportion of knot free and knot containing wood, which is relevant for grading round wood into quality classes according to current grading standards. Therefore detailed scar assessment could be used to achieve important quality information on internal knottiness of complete logs.

The results show that current CT-technology can be used for detailed analysis of knottiness for beech, although there are still technical restrictions like maximal stem diameter. Further development of automated knot detection on CT-images is still needed for beech.

# INTRODUCTION

Wood procurement is a decision under uncertainties, as the quality of timber is not known exactly prior to cutting the wood. However, buyers do estimate the quality considering several indicators. KNOKE ET AL. (2006) identified the currently five most important quality indicators for buyers of beech (*Fagus sylvativa* L.) logs: 1. red heartwood, 2. spiral grain, 3. stem curvature, 4. roughness of the bark, and 5. growth stresses. Information on these indicators can be collected without much effort. Additional information on quality could be gained by assessing branch scars as quality indicators. Former studies could show that the shape of branch scars is correlated to the size of internal knots (SCHULZ 1961). Considering this correlation and the fact that current grading standards for sawn beech timber are regarding knots as one of the most important quality variables (European Standard EN 975-1:2011-08), branch scars could be used to estimate timber quality of logs.

However, obtaining information on wood quality before buying is only profitable if the information gained was cheaper than the profit achieved by having that information available. For logs showing a high variation in price, as it can be expected for beech, more information on timber quality could be profitable (KANGAS ET AL. 2010).

To verify the correlation between branch seal quotient and the size of occluded knots on a large data set, detailed information on log internal features, in particular position and size of knots, is required. Different technologies could be used to determine log internal features. One suitable non-invasive technology is CT scanning (WEI ET AL. 2011).

The objectives of this study were: first, to validate the knot characteristics as they were assessed in CT images and, second, to confirm the correlation between the shape of branch scars and certain knot characteristics.

# MATERIAL AND METHODS

#### Study Objects

For this study 33 beech trees from three different test sites in Baden-Württemberg, SW-Germany, were felled. The trees were of different age, various diameters between 35 and 45 cm, and had different branch and knot sizes and amounts of knots. Thus, the logs were of different quality. After felling, the trees were bucked to length of approximately 4 m and transported to Freiburg.

#### Manual Scar Assessment

On these logs position and size of all branch scars that were visible on the bark were manually assessed. The measured size parameters were the height and width of the seal and the height of the moustache as described by WERNSDÖRFER ET AL. (2005).

#### Acquisition of CT data

"All logs were scanned with the *Microtec CT.LOG* located at the FVA in Freiburg. For the scans a voltage of 180 kV, a current of 14 mA, and a number of 900 views per rotation were used. The resolution in crosscuts was 1.1 mm; for longitudinal resolution 5 mm was chosen. From the raw data a three-dimensional data block was computed, where the grey-value of each voxel (3D-pixel) represents the amount of x-ray absorption and x-ray scattering of the corresponding point in the log." (BAUMGARTNER ET AL. 2010)

#### Analysis of CT Data

The first step of the analysis of the CT images was an automated detection of the pith, followed by a manual knot assessment.

# **Detection of the pith**

"The pith is the origin of every knot and an approximation of the geometrical centre of a log except for logs showing extreme eccentricity. The position of the pith plays a decisive role in the analysis of CT data and, thus, the first step of analysis was the detection of the pith. A modification of the method described by LONGUETAUD ET AL. (2004) was used for the determination of the pith position. This method, derived in principle from the Hough transform, exploits the fact that the pith is supposed to represent the centre of a set of concentric circular structures, i.e. the annual rings, and detects the pixel representing the pith position as the point of maximum intersection of lines in gradient direction in an accumulator array." (BAUMGARTNER ET AL. 2010)

#### **Detection and measurement of knots**

The images were manually examined for irregularities in grey values, representing the knots. Several size and position parameters were assessed (Fig. 1). For every knot the radial distance from the pith to the end of the sound part (LIVETO), the dead part (DEADTO), and the rotten part (LONG3D) was measured. Also the height (A) and width (B) of the knot was assessed every 20-mm-step from the pith. All knots were assumed to be round, so height and width were equivalent to each other. For each of these 20-mm-steps the distance (C) from the centre of the knot to a horizontal line at height of the lowest point of the knot was calculated.

The position of each knot starting point was described by its x-y-zcoordinates. The direction of the knots was described by the azimuth (ALPHA) to a reference line.

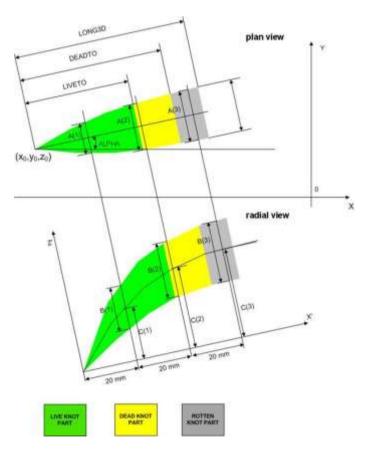


Figure 1: Knot size information assessed in the CT images

# Validation of CT Measurements

The results of the knot measurements in the CT images were checked for accuracy through validation measurements on sawn timber and radial cuts. Twenty logs were cut to boards of 30 mm thickness to assess position and size of all features that were visible on the surfaces. Radial cuts were performed on 26 knots to assess the same size parameters as described for the CT measurements (Fig. 1, lower part).

# Matching Scar and Knot Information

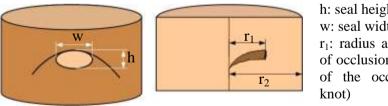
Scar and knot data were matched by comparing the position and spatial orientation of the features. The position was described relative to the butt end of the log and the orientation for knots and scars as the azimuth and a line between the pith and the centre of the seal, respectively.

# **Calculating Correlation**

The ratio between height and width of a scar seal, also known as scar seal quotient, is directly related to the size of the subjacent knot. This correlation is described by Eq. 1 (SCHULZ 1961). The example in Fig. 2 shows a scar seal quotient of 1:2 and the corresponding radial cut, where the ration between the current radius and the size of the occluded knot is also 1:2.

To test this finding a regression analysis was performed on our data by fitting a linear model using the software R (R DEVELOPMENT CORE ТЕАМ. 2011).

$$h / w = r_1 / r_2$$



h: seal height w: seal width r<sub>1</sub>: radius at time of occlusion (size of the occluded r<sub>2</sub>: current radius

Figure 2: Parameters of the scar seal quotient and their correlation to the knot containing core (adapted from: Schulz 1961)

# **RESULTS AND DISCUSSION**

## Validation of CT Measurements

Both, the size of assessed knots in CT measurements and the total number of knots were compared to manual measurements on sawn timber and on radial cuts to check them for accuracy.

The assembly of the knot information assessed on board surfaces was difficult with the available data, as not all features could be related to features on other boards Therefore they could not be traced back to a certain knot. As a consequence, the data validation is largely based on radial cuts.

# **Qualitative validation – Size of knots**

Validation measurements of knot sizes on radial cuts showed high accuracy of the measurements in the CT images (Fig. 2). The mean difference in total length was 7.3 mm with a standard deviation of 5.2 mm.

The reference measurements on radial cuts, however, may in some cases be imprecise, as the cut may not have been positioned at the largest extension of the knot and thus might have led to an underestimation of the total length.

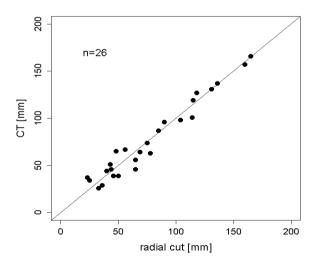


Figure 2:Total knot length measured on radial cuts and in CT images

A general problem in CT image analysis of beech logs are low density differences between knots and surrounding wood and also between different

parts of the knots. As a result of this, it was not possible to distinguish between the dead and the rotten knot part, hence for the analysis both of these parts were described as dead.

The measurements of the extension of the dead knot zone (Fig. 3) show stronger proportional differences than the measurements of the total length (means of 37 % and 14 %, respectively). The mean difference for the dead knot zone is 6.9 mm with a standard deviation of 8.8 mm. One reason for this was, that the optical delineation between sound and dead part in the CT images was difficult through similar grey values. This problem could was more pronounced in logs of larger diameter. The tendency to underestimate the dead knot zone in the images cannot be explained by normal measurement errors and therefore is an effect of the practices of the person doing the manual measurements.

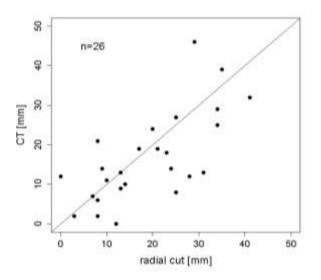


Figure 3: Length of the dead knot zone on radial cuts and in CT images

# Quantitative validation- Number of identified knots

The number of identified knots in CT images strongly depends on the resolution of the scans, as this determines the minimal size of identifiable and measurable knots. Due to the described problems with the data analysis from sawn timber, a comparison with the number of knots found on the boards to the number found in the CT images was not possible. The high accuracy of the length measurements, however, shows that precise measurements are possible. Therefore it can be assumed, that all knots that are relevant for grading purposes can be assessed with the chosen resolution.

# Correlation between Branch Scars and Internal Knots

A linear regression analysis could confirm a strong correlation ( $r^2=0.63$ ) between branch seal quotient and knotty core derived from CT images (Fig. 6).

No clear trend for under- or overestimation could be found, so the measurements do not seem to be biased. Some of the outliers might be caused by scars that have been matched to wrong internal knots.

Knots showing a knotty core > 1 were all non-occluded knots and therefore growing out of the stem. Correct automated radius measurements in some cases failed for such knots, meaning they resulted in a radius smaller than the extension of the knot.

The slope of the fitted linear model is 0.86, so Eq. 1 (which presumes a slope of 1) can be utilised to get a good estimate of the size of occluded knots. For the quality assessment of logs, scar assessment therefore could be useful.

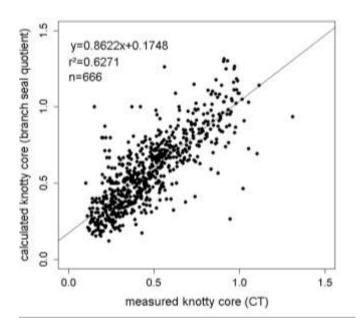


Figure 6: Proportional knotty core compared to the branch seal quotient

# CONCLUSIONS

Results show that the assessment of internal knots is possible for beech, although density differences between knots and surrounding wood are low. Validation measurements on radial cuts could show a high accuracy of manual knot assessment in CT images. Therefore CT-technology is suitable to assess knot characteristics in beech, however automated knot detection still needs further research.

CT-derived knot information could be used to create virtual boards with cutting simulation software. Identified defects could be used to assess the quality of the boards by applying grading standards.

The results of this study could confirm the strong correlation between branch seal quotient and the knot containing core. As a consequence, the assessment of branch scars and the calculation of the seal quotients could be used to derive more detailed information on internal knottiness and thus, wood quality. Depending on the effort needed to assess the branch seal quotient, it could be used as additional quality indicator for beech wood.

# REFERENCES

BAUMGARTNER, R.; BRÜCHERT, F.; SAUTER, U. H. (2010) Knots in CT scans of Scots pine logs. In: Conference Proceedings Final Conference of COST Action E53 "The Future of Quality Control for Wood & Wood Products", 4-7th May 2010, Edinburgh –

http://www.coste53.net/downloads/Edinburgh/Edinburgh-Presentation/45.pdf (29.06.2012)

EUROPEAN STANDARD EN 975-1:2009 + AC:2010 (2011-8) Sawn timber – Appearance grading of hardwoods – Part 1: Oak and beech; German version

KANGAS, A.; MÄKINEN, H.; LYHYKÄINEN, H. T. (2010) Value of quality information of Scots pine stands in timber bidding. In: *Canadian Journal of Forest Reseach*, **40** (9), 1781–1790.

KNOKE, T., STANG, S., REMLER, N., AND SEIFERT, T. (2006) Ranking the importance of quality variables for the price of high quality beech timber (*Fagus sylvatica* L.). Annals of Forest Science, **63** (4), 399-413.

LONGUETAUD, F., LEBAN, J.M., MOTHE, F., KERRIEN, E., AND BERGER, M.O. (2004) Automatic detection of pith on CT images of spruce logs. *Computers and Electronics in Agriculture*, **44** (2), 107-119.

R DEVELOPMENT CORE TEAM (2011) R: A language and environment for statistical computing. Vienna, Austria: R Foundation for Statistical Computing. http://www.R-project.org/.

SCHULZ, H. (1961) Beurteilung der Qualitätsentwicklung junger Bäume. In: *Forstarchiv* **32**, 89–99.

WEI, Q.; LEBLON, B.; LA ROCQUE, A. (2011): On the use of X-ray computed tomography for determining wood properties: a review. In: *Canadian Journal of Forest Reseach*, **41** (11), 2120–2140.

WERNSDÖRFER, H., LE MOGUÉDEC, G., CONSTANT, T., MOTHE, F., SEELING, U., NEPVEU, G., (2005) Approach to the estimation of red heart occurrence in *Fagus sylvatica* based on geometric relationships between branch scar development and knot dimensions, *Scandinavian Journal of Forest Research*, **20** 448–455.

# Roughness of black alder wood surfaces after milling and sanding

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Keywords: black alder, milling, sanding, roughness.

# ABSTRACT

The paper presents a research study upon the roughness of surfaces made of black alder wood after their processing by straight milling and sanding under different processing schedules.

The samples made of black alder wood were processed on their longitudinal edges by straight milling when using a milling cutter (100 mm diameter) with glued straight plates made of CMS, on the vertical milling machine of MNF10 type, endowed with a mechanical feed device. The factorial experiment with three variables (feed speed, cutting depth and cutting width) was used.

The milling was performed by respecting different cutting schedules and the matrix of experiments comprised: rotation speed, feed speed, cutting depth and cutting width.

After each milling process performed according to a certain cutting schedule, longitudinal lamellas were obtained by saw-ripping and they were packed and stored with a view to preserve them and to allow further roughness measurements. The processing roughness was measured along the processing direction with the help of the MicroProf FRT device.

The sanding process was performed on the SANDING MASTER wide belt sander The factorial experiment with two variables (feed speed and cutting depth) was used. The processing by sanding (120 final grit size) was performed by respecting different cutting schedules. Processing direction, feed speed, cutting depth for two wetting phases, with and without wetting, were the variables. The processing roughness was measured perpendicular to the processing direction by using the same optical device. All data were processed by using a non linear regression method and an SPSS variance analysis was then applied to the results of each processing, milling and sanding, in order to establish the influence and the effect intensity of each variable on the surface quality expressed through the roughness parameters.

The study revealed that good longitudinal quality surfaces were obtained after milling when using low feed speed and light cutting depth. Low values for the processing roughness were obtained after parallel sanding and also the wetting did not present significant influence on the sanded surface roughness. This study and its results can be successfully used in wood industry.

# INTRODUCTION

The milling process defined by the literature as "the queen of processing" was studied by many specialists from abroad and Romania as well. Some research works were focused on the processing schedule optimization and special results concerning the surface quality were also obtained (KILIC 2005, VEGA 2005, USTA 2006, SALCA 2008, FOTIN 2009). The optimal cutting schedule with a view to obtain good quality surface has been the topic of previous studies (TARAN 1973) as well.

The sanding process has its main purpose to remove the irregularities from the wood surface caused by previous processes (WILLIAMS AND MORRIS 1998, LIHRA AND GANEV 1999, TARAN 2000). A lot of research projects were carried out on sanding of wood products. Important papers which established the main framework of sanding the wooden surfaces were published. Most of them reffered to the sanded surface quality under the influence of cutting schedules (PAHLITZSCH 1970, POP 1979), applied process through the processing direction and grain orientation (TAYLOR ET AL.1999, CARRANO ET AL.2002), tool features (PAHLITZSCH 1970, POP 1979, COTTA 1982, CARRANO ET AL. 2002, SINN ET AL.2004, DE MOURA AND HERNANDEZ 2006, GURAU 2005, RATNASINGAM AND SCHOLZ 2006) and wood species (COTTA 1982, GURAU 2005, SALONI ET AL.2005, SINN ET AL.2004). Ra, Rz and Rk family were the most used roughness parameters within all these studies. According to SANDAK (2005) and GURAU (2005), Rk and Rpk give the most important reference on the quality of processed wooden surfaces. Within the specialty literature only few data related to the processing of black alder wood were found (MALKOCOGLU 2006) and they referred to the rate of free defect pieces without relevant data value.

The present study is part of a research work focused on black alder wood workability and it deals with the optimization of processing by milling and sanding with a view to achieve the wood species capitalization in furniture manufacturing (SALCA 2008). The objective of this study was to evaluate the surface quality of black alder specimens as function of milling and sanding processes under different cutting schedules.

# **EXPERIMENTAL METHODS**

#### Material and method

The samples made of black alder wood were processed on their longitudinal edges (1000 mm long at 8% moisture content) by straight milling when using a milling cutter (100 mm diameter) with glued straight plates made of CMS, on the vertical milling machine of MNF10 type, endowed with a mechanical feed device. The factorial experiment (LAURENZI 2000) with three variables, namely: five feed speeds (4.5, 9, 13.5, 18 and 22.5 m/min), five cutting depths (1, 2, 3, 4 and 5 mm) and five cutting widths (20, 25, 30, 35 and 40 mm) was used. According to the algorithm 20 specimens were used. The processing by milling was performed by respecting different cutting schedules and the matrix of experiments under study comprised the following parameters: rotation speed, feed speed, cutting depth and cutting width, as shown in Table 1. After each milling process performed according to a certain cutting schedule, longitudinal lamellas were obtained by sawripping and they were packed and stored with a view to preserve them and to allow further roughness measurements.

The sanding process was performed by using free defect samples with the dimensions of about 300 x 95 x 16 mm and 8% moisture content on the SANDING MASTER wide belt sander at NIKMOB Company from Nehoiu. The equipment has pneumatic oscillation sytem along with self cleaning setup. Three types of abrasives (60, 100 and 120 grit size by respecting this sequence) manufactured from corundum abrasive were used for the sanding process. Initially 60 grits sandpaper was used for calibration purpose. The factorial experiment (LAURENZI 2000) with two variables, namely: five feed speeds (4, 8, 12, 16 and 20 m/min) and five cutting depths (0.1, 0.2, 0.3, 0.4 and 0.5 mm) was used. 39 specimens, 3 sets of 13 samples each one for the three processing directions were used according to the algorithm. The processing by sanding (using a certain order of grit sizes: 60, 100 and 120) was performed by respecting different cutting schedules obtained with the

following variables: processing direction, feed speed, cutting depth for two wetting phases. Table 1 presents the matrix of experiments under study. All samples were wetted on halfway of their surfaces, before each pass, to analyse effect of the wetting on the roughness of sanded surfaces. Frames were used to determine and respect the sanding direction (parallel, perpendicular and at 45 degrees angle to the wood grain orientation).

Table 1: Maintx of experiments under study								
	Straight milling process			Sanding process				
Machine		MNF 1	0		SANDING MASTER			
Tool	millin	g cutter (100 i	nm diamet	er)	Abrasives (60, 100 and 120 grit size)			
					II (Parallel to the wood grain orientation)			
D					$\perp$ (Perpendicular to the wood grain			
Processing direction	longitudinal				orientation)			
direction					45° (at 45° angle to the wood grain			
					orientation)			
Number of samples and dimensions	20 spec	eimens	1000 mm length		39 specimens 30		0 x 95 x 16 mm	
	Rotation	Feed	Cut	ting	Wetting Feed speed Cutting d			
Processing	speed [rot/min]	speed [m/min]	depth [mm]	width [mm]	Wetting	feed [m/r		Cutting depth [mm]
parameters	6620 9732	9 13.5 18	1 2 3	30	with without	8 1 1		0.1 0.2 0.3

#### Table 1: Matrix of experiments under study

# **Roughness measurement**

A profilometer of MicroProf FRT type was the device used for roughness measurement. This device is a new standard instrument used for optical evaluation of surface roughness.

The scanning parameters are presented in Table 2. It is worth to be mentioned that the evaluation length, sampling length and resolution were selected according to the recommendations for wooden surfaces given by Gurau (2005). With a view to respect the roughness measurement direction as already presented, some specific wooden devices were used. One roughness measurement were performed along the processing direction for milled specimens and in the case of sanded surfaces, two roughness measurements were performed per each sample, on areas with and without wetting before sanding.

Table 2. Microfroj FKT Scanning parameters					
Scanning set parameters	Parameter value				
Scanning mode	2D				
Scanning speed	750 μm/s				
Number of points per line	10000 points				
Evaluation length	50 mm				
Sampling length	2.5 mm				
Resolution	5 µm				
Direction of measurement	- Along the processing direction for milled surfaces				
Direction of measurement	- Perpendicular to the processing direction for sanded surfaces				

Table 2: MicroProf FRT Scanning parameters

Two parameters from the *Rk* family (*Rk*, *Rvk* şi *Rpk*), namely *Rk* şi *Rpk*, were selected according to ISO 13565-2: 1996 standard. *Rvk* was excluded because the anatomical roughness was not removed. The *Rk* parameter, defined as the roughness core depth, is proposed by Gurău (2005) and Sandak (2005) as the most representative indicator of processing roughness (Table 3).

Table 3: Roughness parameters

Roughness parameter	Description	Representation		
<i>Rk</i> Family, µm	- the profile peaks area <i>Rpk</i> (parameter for the fuzzy grain evaluation); - the central profile area, <i>Rk</i> (parameter for the processing roughness evaluation); - the profile valleys area, <i>Rvk</i> (parameter for the anatomical roughness evaluation)	R <sub>pk</sub> Augeochegenate R <sub>vk</sub> R <sub>vk</sub> M <sub>r1</sub> M <sub>r2</sub>		

#### Processing of data

The profilometer software allowed to display the surface topography under study. The profile roughness analysis was also performed and the roughness profile was obtained after a previous data filtering with the Gaussian filter, automatically applied. All data were processed by using a non linear regression method by respecting an equation of 2<sup>nd</sup> degree type with three variables in the case of milled surfaces and two variables for sanded surfaces. The results for each processing were analysed by studying and interpreting the specific segments of modelling in correlation with those from industrial conditions and according to the specialty literature. Thus, discussing the milling process, some extreme values were removed from the initial study according to the literature (TARAN 1973) and due to the restrictive representation during the real experiment. Three feed speeds (9, 13.5 and 18 m/min) and three representative cutting depths (1, 2 and 3 mm) only for a cutting width of about 30 mm, were analysed. The cutting width does not influence the quality of processed surfaces, but it has an important

impact upon the dynamic elements of the milling process and thus it presents an indirect influence upon surface.

In the case of sanded surfaces the same restrictive approach was carried out as follows: the experimental work was well represented for three feed speeds, such as 8, 12 and 16 m/min, (optimal values from 8 to 25 m/min) and three cutting depths (0.1, 0.2 and 0.3 mm), pointed as optimal for sanding solid wood samples with grit sizes over 100 (TARAN 2000).

An SPSS variance analysis was then applied in order to establish the influence and the effect intensity of each variable and groups of variables on the surface quality expressed through the two processing roughness parameters, Rk and Rpk respectively.

# **RESULTS AND DISCUSSION**

Comparative graphically representations were achieved for the two roughness parameters under study depending on the matrix of experiments for each one of the two processes, milling and sanding, respectively. Figure 1 presents the variation of Rk and Rpk parameters on the surfaces processed by longitudinal straight milling with two rotation speeds (6620 and 9732 rot/min), depending on the feed speed and cutting depth for a cutting width of about 30 mm.

The representations revealed that once the feed speed and cutting depth increased when processing by milling with the rotation speed of about 6620 rot/min, the two roughness parameters respected an increased trend, compared to the situation when processing with the rotation speed of about 9732 rot/min, when a light increase was noticed for the processing roughness parameter, *Rk*. The best surface quality was achieved after processing by milling with a rotation speed of about 6620 rot/min, and a feed speed of about 9 m/min for 1mm as cutting depth (*Rk*=12.4  $\mu m$  and *Rpk*=5.8  $\mu m$ , respectively).

FOTIN (2009) obtained low values for Rk, ranging from 10 to 12  $\mu m$  for birch wood when milled with similar cutters. In the case of milled surfaces, the SPSS analysis of variance applied to all values of Rk parameters showed a significant cumulative effect (*Sig.* 0.016<0.05) for two factors under grouping approach, namely the rotation speed and feed speed. The relation intensity is highlighted by the value of  $\eta^2$  coefficient (0.873 >0.5) which indicates an important interaction of those two factors upon the processing roughness parameter Rk. The same factors significantly influenced the parameter for the fuzzy grain evaluation, Rpk, when *Sig.*=0.029<0.05 and the relation intensity was given by  $\eta^2$  coefficient, 0.828>0.5.

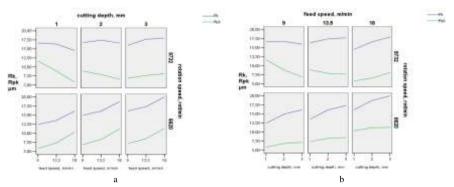


Figure 1: Variation of roughness parameters, Rk and Rpk, depending on the feed speed for each value of the cutting depth (a) and depending on the cutting depth for each value of the feed speed (b) after processing by longitudinal milling for a cutting width of about 30 mm at two rotation speeds (6620 and 9732 rot/min)

The variations of the two roughness parameters, *Rk* and *Rpk*, determined on sanded surfaces processed under different cutting schedules, depending on the processing direction, feed speed, cutting depth, with and without wetting before sanding are presented in Fig. 2. The same general increased trend was noticed for both roughness parameters once the feed speed and cutting depth increased for all the three processing directions, namely parallel, perpendicular and at 45 degrees angle to the grain orientation. The values recorded for Rk si Rpk roughness parameters are lower in the case of perpendicular processing, but this processing produces less aesthetic surfaces and thus it is not recommended. It was also noticed that the wetting phase is not necessary with a view to obtain good sanded surfaces. The best surfaces were obtained when sanding without wetting for the feed speed of about 8 m/min and the cutting depth of about 0.1 mm. When processing at 45 angle and parallel to the wood structure orientation, as resulted from Fig.2, the Rk and Rpk values were of about 22.3 and 19.6 µm and 6.4 and 6.6 µm, respectively. Birch and oak surfaces sanded with similar grit size were evaluated by FOTIN (2009) and GURAU (2005), respectively. Rk values for birch and oak were 15.5  $\mu m$  and 7.6  $\mu m$ , respectively, which confirm that the higher the wood density, the better the surface quality is.

In the case of sanded surfaces the SPSS analysis of variance presented the significant cumulative effect of three factors, processing direction - feed speed – wetting, upon the *Rk* roughness parameters expressed by *Sig.*=0.014<0.05 coefficient when their relation intensity was very strong ( $\eta^2$ =0.759>0.5). Two pairs presented a similar cumulative effect and a strong relation intensity upon the parameter for fuzzy grain evaluation, *Rpk*.

They were: processing direction - feed speed with Sig.=0.047<0.05 and  $\eta^2$ =0.795>0.5 and feed speed – cutting depth with Sig.= 0.013<0.05 and  $\eta^2$ =0.843>0.5.

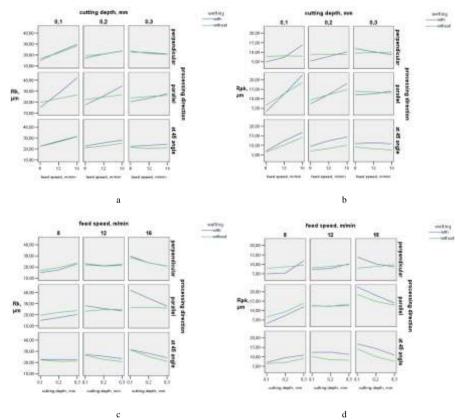


Figure 2: Variation of roughness parameters, Rk and Rpk, depending on the feed speed for each value of the cutting depth (a and b) and depending on the cutting depth for each value of the feed speed (c and d) after processing by sanding with a three grit sizes program (60, 100, 120) and respecting three processing directions

#### CONCLUSIONS

The study revealed that good longitudinal quality surfaces were obtained after milling when using low feed speed and light cutting depth. They appeared better than sanded surfaces. The rotation speed and feed speed presented the most intense relation, having the most significant cumulative effect upon both roughness parameters under study. Low values for the processing roughness were obtained after parallel sanding and also the wetting did not present significant influence on the sanded surface roughness. The processing direction, feed speed and wetting presented the greatest significant cumulative effect on the processing roughness.

This study and its results can be successfully used in wood industry. It is also a challenge to impel the interest of the specialists from the wood processing department through this hardwood species, less studied and somehow ignored for a long period but with a special appearance and a high potential of use.

#### REFERENCES

CARRANO, A.L, TAYLOR, R. AND LEMASTER, R. (2002) Parametric characterization of peripheral sanding. *Forest Products Journal*, 52 (9) p.44-50.

COTTA, N., NĂSTASE, V. AND POP, I. (1982) *Şlefuirea lemnului şi* peliculelor de acoperire. Editura Tehnică București.

DE MOURA, L.F. AND HERNÁNDEZ, R.E. (2006) Effects of abrasive mineral, grit size and feed speed on the quality of sanded surfaces of sugar maple wood. *Wood Science and Technology*, 40(6), 517-530.

FOTIN, A. (2009) Contributions to the optimization through milling and sanding of birch wood with a view to use it in furniture manufacturing ad ther wooden products. PhD Thesis. Transilvania University of Brasov.

GURAU, L., WILLIAM, M. AND IRLE, M. (2005) Processing roughness of sanded wood surfaces. *Holz als Roh und Werkstoff*, 63(1), 43-52.

KILIC, M., HIZIROGLU, S., AND BURDULU, E. (2005) Effect of Machining on Surface Roughness of Wood. *Building and Environment*, DOI: 10.1016/j.buildenv.2005.05.008.

LAURENZI, W. (2000) Contributions to the Modelling and Optimization of Wood Cutting by Circular Saws with a View to Control the Process by Using Computer-Aided System. *PhD Thesis*, Transilvania University of Brasov.

LIHRA, T. AND GANEV, S. (1999) Machining properties of eastern species and composite panels. Forintek Canada Corp., Division de l'Est, Ste-Foy, Québec, 62 p.

MALKOCOGLU, A., AND OZDEMIR, T. (2006) The Machining Properties of Some Hardwoods and Softwoods Naturally Grown in Eastern Black Sea Region of Turkey. *Journal of Materials Processing Technology*, No. 173, 315-320.

PAHLITZSCH, G. (1970) International state of research in the field of sanding. *Holz als Roh-und Werkstoff*, 28(9), 329-343.

POP, I. (1979) Contribuții la studiul calității suprafețelor prelucrate prin șlefuire. *Industria Lemnului*, Nr.1.

RATNASINGAM, J. AND SCHOLZ., F. (2006) Optimal surface roughness for high-quality finish on rubberwood Hevea brasiliensis . *Holz als Roh-und Werkstoff*, 64 (4), 343-345.

SALCA, E. (2008) Contributions to the optimization of alder wood processing by milling and sanding in order to achieve its capitalization in furniture manufacturing. *PhD Thesis*.Transilvania University of Brasov.

SALONI, D., LEMASTER, R. AND JACKSON, S. (2005) Abrasive machining process characterization on material removal rate, final surface texture and power consumption for wood. *Forest Products Journal*, 55(12), 35-41.

SANDAK, J., AND MARTINO, N. (2005) Wood Surface Roughness-What Is It. In: *Proceedings of 17th International Wood machining Seminar*, Rosenheim, Germany, Vol.1, pp. 242-250.

SINN, G., M. GINKL, REITERER, A. AND STANZL-TSCHEGG., S. (2004) Changes in the surface properties of wood due to sanding. *Holzforschung*. 58 (3), 246-251.

TARAN. N. (1973) Roughness of Milled Surfaces, Main Criterion Used when Selecting Optimal Cutting Schedules. *Journal of Wood Industry*, No. 10.

TAYLOR, J., CARRANO, A. AND LEMASTER, R. (1999) Quantification pf process parameters in a wood sanding operation. *Forest Products Journal*, 49(5), 41-46.

ŢĂRAN, N. (2000) Maşini –unelte şi utilaje moderne pentru şlefuirea suprafeţelor lemnoase. Editura LUX LIBRIS Braşov.

USTA, I., DEMIRCI, S., AND KILIC, Y. (2006) Comparison of Surface Roughness of Locust Acacia and European Oak in Terms of the Preparative Process by Planing. *Building and Environment*, DOI: 101016/j.buildenv.2006.07.026.

VEGA, M., AND AGUILERA, A. (2005) Surface Roughness and Cutting Power for Machining Radiata Pine. *Bosque*, 26(1), 101-108.

WILLIAMS, D. AND Morris, R. (1998) Machining and related mechanical properties of 15 B.C. wood species. Forintek Canada Corp., Division de l'Ouest, Vancouver, B.C., 31 p.

\*\*\*ISO13565-2. 1996. Geometrical product specification. Surface texture. Profile method.

Part 2. Height characterization using the linear material ratio curve.

# Suitability of stress wave and electrical resistivity tomography for ring-shake detection in standing Sweet chestnut trees

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**Keywords:** ring-shake, Sweet chestnut (*Castanea sativa*, Mill.), non destructive methods, silvicultural treatment

# ABSTRACT

Sweet chestnut as a species adapted to climate change in Germany has attracted a lot of interest in forestry. A problem for the utilization of the wood is internal defect called ring shake which is not noticeable while the tree is standing and reduces the value of the wood dramatically. It was investigated if two non-destructive methods (stress wave and electrical resistivity tomography) can be used to detect ring shake in standing trees. 55 trees were selected in six stands of the forestry district Haardt in Rhineland-Palatinate, Germany and classified according to three different silvicultural treatments (coppice, low thinning and future tree selection) and two age classes. All of them were measured with the stress wave and the electrical resistivity tomograph. After felling wood moisture content was measured over the stem cross-section.

Stress wave and electrical resistivity tomograms of 46 trees could be compared to stem disks after harvest. The combined application of the two non-destructive methods allowed the detection of ring shake. 16 of 55 trees were affected by ring shake. The highest proportion of affected trees is found in the oldest stand with future tree selection. The wood moisture distribution within the stem cross-section had no significant effect on the proportion of trees with ring shake. Thus, tomography could be used successfully to assess the frequency and distribution of ring shake in standing Sweet chestnut trees and to analyse the effects of silvicultural treatments on the ring shake occurrence.

# INTRODUCTION

Due to global change drought-resistant tree species like Sweet chestnut become interesting for forestry in Germany. Not only the fruits but also the wood is used in many ways especially in outdoor applications. But the wood is affected by a wood defect called ring shake, a crack tangential to annual rings, which reduces the market value dramatically (FONTI and GIUDICI 2001, TRAUTH 2008, FONTI 2002). Hence it is important to analyse its influencing factors and methods to detect the defect already in standing trees for early selection.

# **EXPERIMENTAL METHODS**

#### Stands

55 trees in six stands (A-F) in two age classes (younger and older) with three different silvicultural treatments were randomly selected (Table 1). Climate and soils of the sites were similar. A minimum diameter at breast height (DBH) was set to 25 cm. Silvicultural treatments were:

- Marginal thinning, nearly natural forest
- Coppice, short rotations, many shoots
- Future tree selection, crown exemption

Stand	Tree number	Age [years]	Silvicultural treatment	Mean DBH [cm]
Stand A	01-10	63	Natural forest	38,35
Stand B	11-20	27	Coppice	26,88
Stand C	21-30	39	Natural forest	26,93
Stand D	31-40	64	Coppice	34,73
Stand E	41-45	24	Future tree selection	26,00
Stand F	51-60	120	Future tree selection	45,50

Table 1: Characterization of the sample collectives in the forestry district Haardt

# Experimental setup

Every randomly selected tree was surveyed with a stress wave tomograph (PiCUS<sup>®</sup> sonic tomograph, argus electronic GmbH, Rostock, Germany) and an electrical resistivity tomograph (PiCUS Treetronic<sup>®</sup>, argus electronic, Rostock, Germany) at 1.3 m height. After harvested, wood moisture was measured at 10 evenly spaced points along a radial gradient across a section

at the stem base. Following the stem was cut at measuring plane and the ring-shake occurrence was documented.

# Stress wave tomography

Stress wave tomography for trees (RUST 2008) uses many sonic sensors around the circumference of the tree recording the transmission time of sonic signals simultaneously. Tomograms are reconstructed from apparent relative sonic velocities. Analysis of tomograms is based on the ability of the wood to transmit stress waves (HAABEN et al. 2006) rather than velocity or density maps. In the resulting tomogram (Fig. 1), dark colours like black and brown indicate wood with good sonic-conduction properties. Blue and violet mark areas with relatively low apparent stress wave velocities, indicative of wood defects.

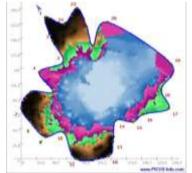


Figure 1: An exemplary stress wave tomograph

# Electrical resistivity

Electrical resistivity tomography mainly visualises chemical properties like ion and moisture content (BIEKER and RUST 2010, BIEKER et al. 2010). The instrument uses point-like electrodes at the boundary of the object (Fig. 2). At two of these a current is injected. The resulting electric field depends on the resistivity distribution and is measured by using the other electrodes pair-wise to obtain a potential difference. Data collection is followed by the tomographic re-construction of the resistivity distribution (Fig. 3). Areas with good conduction are coloured blue, low conductive areas are shown red.

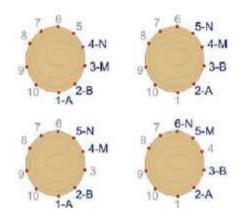


Figure 2: Scheme of supplying current during the electrical resistivity measurement (PiCUS Treetronic® manual, 2008)

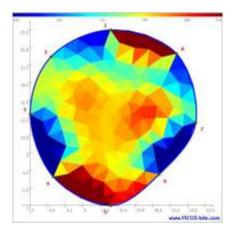


Figure 3: An exemplary electrical resistivity tomograph

#### Moisture measurements

On every harvested stem the moisture content was measured 10 times along a north-south transect. The used instrument (GANN Hydromette RTU 600) measures the moisture via electrical resistance which correlates with the water content in the wood.

# **RESULTS AND DISCUSSION**

#### Results

16 of 55 trees or 29% were affected by ring-shake. The highest proportion was found in stand F, the oldest stand with future tree selection. Details are given in Table 2, those of the other stands in Table 3.

Table 2: Detai	iea informai	tion of the ring	g-snake occurrence in stana F
Tree number	<b>Ring-shake</b>	Length (mm)	Comment
F51	yes	239	no features
F52	yes	160	Ring-shake up to 2,8 m height
F53	yes	270	Ring-shake up to 3,3 m height
F54	yes	190	Ring-shake up to 2,8 m height
F55	yes	75	Ring-shake up to 2,3 m height
F56	yes	100	no features
F57	yes 2	120;150	from 0,35 m to 1,3 m height
F58	yes	70	from 0,50 m to 1,0 m height
F59	yes	not available	from 0,35 m to 1,0 m height
F60	no		

Table 2: Detailed information of the ring shake occurrence in stand F

Table 3: Ring-shake occurrence in the stands A to E

Stand	Count of trees with ring- shake
Α	1 of 10
В	1 of 10
С	2 of 10
D	3 of 10
E	0 of 5
F	9 of 10

Only 46 measurements could be completed due to lack of time. On the tomograms the ring-shake is illustrated as a black line which is drawn by using photos of stem disks out of the measuring plane (shown in Fig. 4). For the correct view the tomographs had to be mirrored.

The stress wave tomogram of tree D 35 (Fig. 5) shows very low apparent relative sound velocities in the area of the ring-shake. This indicates that there is a defect. That it is not a rot is shown by the electrical resistivity tomogram (Fig. 5): it does not show low electrical resistivity as it would be typical, but a rather dry area at the ring-shake.

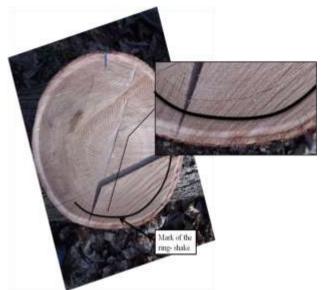


Figure 4: Principal of drawing ring-shake in the tomographs

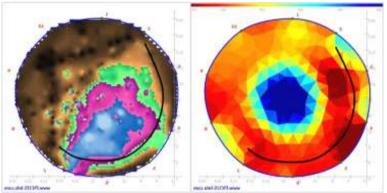


Figure 5: Stress wave and electrical resistivity tomograph of sample tree D 35

In addition to the visual analysis of the tomographs, scattergrams were established with the statistic program "R" to detect special patterns of sonic velocities and electrical resistances (Fig. 6). The x-axis shows the mean sonic velocity in m/s, the y-axis is the mean electrical resistance in  $\Omega$ m. Ring-shake affected trees are written in light blue, others in orange. The type size gives information about the skewness of the distribution of the electrical resistances. It is conspicuous that trees with ring-shake show higher velocities above 1050 m/s. They concentrate between 915 and 1050 m/s.

The electrical resistance lies between 500 and 700  $\Omega$ m in which ring-shake affected trees are spread less. Furthermore trees with ring-shake show a left-skewed distribution of the electrical resistances with values from zero and below visible by the bigger type size. All trees with ring-shake are below zero, whereas trees without ring-shake show values below as well as above zero.

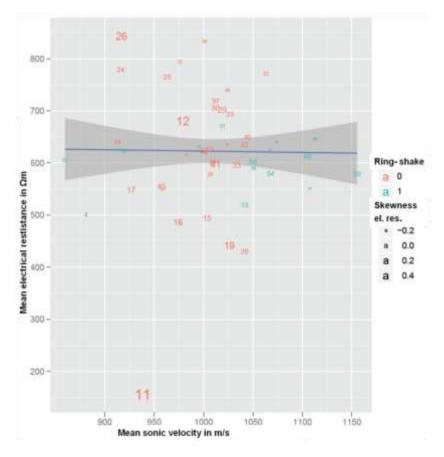


Figure 6: Scattergram of sonic velocity, electrical resistance and skewness

Fig. 7 shows the moisture content along the gradient from the pith to the sapwood of the sample trees for every stand. On the x-axis is shown the radial gradient from the pith to the sapwood following measuring point I to V, on the y-axis is shown the measured moisture in percentage. It is conspicuous that the moisture content in the sapwood is higher than in the heartwood of the cross-section.

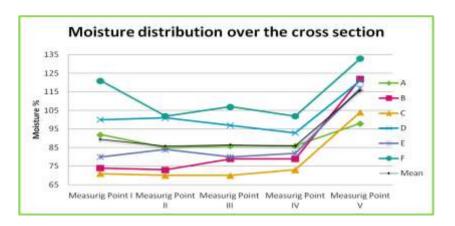


Figure 7: Line plot of the moisture distribution over the sample tree cross sections in all investigated stands.

# Discussion

# **Ring-shake detection**

The resolution of the tomograms was low because of the use of eight sensors per tree only. This low resolution most likely concealed ring-shake in some stress wave tomograms. Only in some cases they show signs of ring-shake. This leads to the conclusion that stress wave tomography must be applied with a higher spatial resolution for ring-shake detection. It is notable that trees with ring-shake show higher mean sonic velocities. Applied with more sensors and combined with electrical resistivity tomography to differentiate between decay and ring-shake, stress wave tomography will have a higher detection rate.

# Factors of influence

As seen in Figure 7 the stands F and C show very different moisture contents and distributions. On the one hand sample trees in stand F show high moisture content and an alternating distribution, on the other hand sample trees in stand C show lower moisture content and a homogenous distribution. There was no statistical correlation between ring-shake and wood moisture content.

The trees in stands E, B and C have a similar DBH but are of different age. The youngest trees (24 years) in stand E are not affected by ring-shake whereas in stand B (27 years) ring-shake affects 10 % of the sampled trees.

The trees in stand C are 39 years old and show a ring-shake proportion of 20 %. So ring-shake occurs significant more often with increasing age. Furthermore it is remarkable that in older trees the ring-shake extends more in height of the stem.

The trees in stands E (24 years) and B (27 years) are nearly in the same age and DBH. Stand B has a ring-shake proportion of 10 %, but stand E has 0 % ring-shake. The two stands had been treated differently: stand B is a coppice area whereas stand E is an area with future tree selection. A similar example is a comparison between stand A (63 years) and D (64 years) which are in similar age and DBH but differ in their silvicultural treatment: stand A is a nearly natural forest and 10 % of the sample trees were affected by ringshake. Stand D is a coppice area and has a ring-shake proportion of 30 %. Summing up the silvicultural treatment seems to influence the proportion of trees with ring-shake occurrence and should be investigated more intensive.

# CONCLUSIONS

- 1. Ring-shake can be detected with a combination of stress wave and electrical resistivity tomography.
- 2. Ring-shake incidence increases significantly with age of the tree.
- 3. The influence of silvicultural treatments cannot be determined because of the small sample size.
- 4. The moisture distribution over the stem cross section does not correlate with ring-shake.
- 5. The resolution of the tomograms was chosen to low and must be increased in future studies.
- 6. More potential factors of influence should be investigated.
- 7. Other non-destructive methods could be tested for ring-shake detection to get more secure results.

# ACKNOWLEDGEMENT

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#### REFERENCES

BIEKER, D., KEHR, R., WEBER, G., RUST, S. (2010) Non-destructive monitoring of early stages of white rot by Trametes versicolor in Fraxinus excelsior. In: *Annals of Forest Science*, 67, p. 210

BIEKER, D., RUST, S. (2010) Electric resistivity tomography shows radial variation of electrolytes in Quercus robur. In: *Canadian Journal of Forest Research*, 40, pp. 1189–1193

FONTI, P. (2002) Investigation into ring shake of chestnut. Graduate thesis at ETH Swiss Federal Institute of Technology in Zürich

FONTI, P.; GIUDICI, F. (2001) Quantitative and qualitative recording of wood volume from an over-aged chestnut coppice forest In: *Schweizerischer Forstverein, Zürich, Switzerland, Schweizerische Zeitschrift für Forstwesen*, 152, 10, pp 417 – 424

HAABEN, C.; SANDERS, C.; HAPLA, F. (2006) Untersuchung der Stammqualität verschiedener Laubholzarten mittels Schallimpuls-Tomographie In: *Holztechnologie*, 47: 6, pp. 5-12

RUST, S. (2008) Geräte und Verfahren zur eingehenden Baumuntersuchung In: ROLOFF, A. (2008) Baumpflege. Ulmer Verlag, Stuttgart. Slightly revised version

TRAUTH, A. (2008) Bedeutung der Esskastanie in Deutschland unter Berücksichtigung des prognostizierten Klimawandels. Diploma thesis at HAWK Göttingen

# Colour characterization of various hardwoods

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**Keywords:** Wood colour characteristics, CIE L\*a\*b\*-colour system, colour intensification (German: anfeuern)

# ABSTRACT

Wood as a material absorbs and reflects the visible light and this physical interaction produces the typical wood colour within a range of almost white, various yellowish, reddish and brownish hues to almost black. The colour characteristics depend on the chemical components of wood that interact with the light which has been analyzed in many studies. Lots of scientific research has been performed on the discolouration of wood surfaces caused by light irradiation and technological processes such as wood drying, steaming or modification. There is hardly any reproducible digital data on the wood colour of the various wood species as in most wood handbooks the colour of the various wood species still is described as "almost white", "bright yellow", "reddish-brown", "dark brown" etc.

In our current research we set a focus on a spectrophotometric characterization of various wood species, especially hardwood species by applying the CIE-L\*a\*b\*-colour-system on 17 domestic wood species. According to an extensive literature review and our own experience, colourimetry is a good method for obtaining objective information on the colour of wood. A special focus was put on the analysis of colour enhancement (German: "anfeuern") by wetting (clear lacquering) a wood surface. It could be shown how the various wood species react to wetting by a colour enhancement analyzed by the CIE-L\*a\*b\*-colour-system. These data also support the understanding of the mechanisms of the colour enhancement as a physical phenomenon of light reflection and refraction on a "dry" and rough surface and a wet or clear coated glazed surface.

Especially the huge variety of hardwood species benefits from the colour enhancement for a more intensive colour appearance which can be capitalized in the furniture and flooring industry. Understanding this colour enhancement also supports innovative natural colour engineering of specific wood species.

#### INTRODUCTION

When visible light strikes an object, all of the light can be reflected or absorbed or certain wavelengths are absorbed and the rest is reflected. The latter case is typical for most of the materials and based on the reflected wavelengths we recognize a certain colour of the material. Wood as a material absorbs and reflects the visible light and this physical interaction produces the typical wood colour within a range of almost white, various yellowish, reddish and brownish hues to almost black. The colour characteristics depend on the chemical components of wood that interact with the light which has been analyzed in many studies such as Hon et al. (2001). Lots of scientific research has been performed on the discolouration of wood surfaces caused by light irradiation and technological processes such as wood drying, steaming or modification (Tolvai, et al. 1995, Hon et al. 2001, Müller et al. 2003, Mitsui 2004, Tolvaj et al. 2005). Oltean et al. (2008) tried to establish a classification of wood surface discolouration due to indoor UV light irradiation by analyzing the dynamics of discolouration and building three groups of wood species with strong, medium and low discolouration.

Another approach to characterize the effect of light on lacquered surfaces as analyzed by Luljka (1997), Forsthuber et al. (2011) and Forsthuber et al. (2012).

Wood colour research was initiated in Japan by Onodera (cit. in Sullivan 1966), who related visual phenomena to spectrophotometric measures of the Y tristimulus value, luminance. An extensive review on the historic development of wood colour measurement is provided by Sullivan (1966). But still today there are hardly any reproducible digital data on the wood colour of the various wood species themselves and in most wood handbooks colour terms of the various wood species are still terms such as "almost white", "bright yellow", pale straw-buff", "reddisch-brown", "darkbrown" etc. (e.g. Wagenführ 1996) rather than distinctive numeric values. Nishino et al. (1998) measured the colour of 97 wood species from French Guiana with a colourimeter according to the CIELAB system. In our current research we set a focus on a spectrophotometric characterization of various wood species, especially hardwood species by applying the the CIE-L\*a\*b\*-colour system

on 24 domestic wood species. According to an extensive literature review and our own experience, colourimetry is a good method for obtaining objective information on the colour of wood. A special focus within this study was put on the analysis of colour enhancement ("anfeuern" in german) by wetting a wood surface (fig 1).

Any wetting (and clear coating) of wood surfaces leads to an intensification and enhancement of the colour. Meichsner et al. (2011) describe the different colour appearance of a dry (uncoated) and a wet (clear coated) wood surface by the microscopic roughness of the respective surface and their reflection characteristics. Due to the laws of reflection less light is reflected from a laquered surface as a part of the diffuse light is "captured" within the laquered (or wet) layer of the surface and is transformed into heat. Therefore a laquered surface exhibits a higher chroma and a lower lightness.



Figure 1: Selection of domestic hardwood species: The upper part shows a sanded surface with no further treatment. The lower part shows colour intensification (German: anfeuern) due to a clear laquer treatment

## MATERIAL AND METHODS

17 domestic hardwood specimens were taken from 20 (in a few cases from 17) different wood specimen collections, where the wood specimen were manufactured from technically kiln dried boards. The specimens with a size of about 100 x 200 mm and a thickness of about 12 mm were conditioned at 20° C and 65% r.H. which resulted in an EMC of about 10 to 13%. Before colour measurement the surfaces were sanded with a sanding paper of a grid of 400 as the last sanding step.

The basis for colour designation lies in a measure of the energy distribution of light reflected from a surface. The spectrometer accomplishes this by selecting any particular wavelength in the light sources and measuring the radiant flux of the selected wavelength that is reflected from a standard surface. This is compared arithmetically to the flux of the same wavelength that is reflected from the test material.

Colour was measured with a spectrophotometer (Phyma CODE 400) at the range of wavelengths between 400 and 700 nm. The light source was defined by a standard illuminant D65 with an observer of  $2^{\circ}$ . The diameter of the field of view was 20 mm and the analysis of the colour data was due to the CIE L\*a\*b\* colour space (EN ISO 11 6664). The CIELAB colour space can also be used for computing the colour difference ( $\Delta E$ ) by computing the Euclidean distance between two points representing the colour stimuli in the colour space (Urland 1999).

Colour measurement was firstly performed on dry untreated surfaces. Afterwards the specimen was put into a pot of distilled water for about 15 seconds and the wetted surface was measured again.

## **RESULTS AND DISCUSSION**

The results of the colour measurement of the untreated native surface and the wetted surface are summarized in table 1.

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$\mathbf{SA}$	74,2	12,0	26,5	29,5	66,0	64,1	17,5	41,6	45,2	67,4	-10,1	5,5	15,1	15,7	1,4	185,0
РО	88,7	5,3	23,0	23,6	76,9	85,4	6,2	31,2	32,0	78,6	-3,3	1,0	8,2	8,4	1,7	8,9
CT	73,5	7,8	28,0	29,1	74,4	65,2	11,9	39,8	41,6	73,4	-8,3	4,1	11,9	12,4	-1,0	15,1
PR	67,2	12,5	30,5	33,0	67,8	57,8	18,8	49,3	49,5	61,9	-9,4	6,3	18,8	16,5	0,0	21,9
ΡY	60,3	16,4	27,7	31,9	59,7	41,3	29,5	61,1	68,3	64,1	-19,0	13,1	33,4	36,3	4,3	40,6
AC	87,9	5,3	22,3	23,0	76,6	83,7	7,4	31,5	32,7	77,1	-4,2	2,0	9,2	9,8	0,6	10,3
ΤI	83,3	6'9	25,2	26,1	74,7	77,5	9,6	37,5	38,7	75,8	-5,8	2,7	12,3	12,6	1,1	13,9
CP	81,2	4,8	22,9	22,8	77,8	75,4	7,4	34,4	35,2	77,8	-5,9	2,6	11,5	12,3	-0,1	13,2
UL	67,1	10,4	27,2	29,0	69,2	57,5	16,3	42,2	45,3	68,9	-9,6	6,0	15,0	16,3	-0,3	18,8
JG	57,4	10,9	24,7	27,1	66,1	39,5	21,1	49,9	54,4	67,0	-18,0	10,1	25,2	27,4	0,9	32,6
QU	67,3	8,8	29,8	31,1	73,4	58,5	13,8	44,9	47,2	72,9	-8,8	5,0	15,1	16,1	-0,6	18,2
вт	80,7	7,8	25,3	25,7	73,0	75,9	10,2	36,0	37,6	74,4	-4,8	2,4	10,7	12,0	1,4	11,9
AL	76,5	10,5	26,2	28,2	68,2	68,1	16,0	42,6	45,5	69,5	-8,3	5,5	16,4	17,3	1,3	19,2
SO	64,7	16,9	30,0	33,3	60,4	52,9	26,3	51,1	57,8	62,4	-11,8	9,4	21,1	24,5	2,0	25,9
RO	60,3	13,3	37,4	39,5	71,1	53,7	18,0	56,6	59,5	72,3	-6,6	4,7	19,3	20,0	1,3	20,9
FA	75,4	10,1	23,7	25,7	67,5	68,3	14,5	36,4	39,2	68,3	-7,1	4,5	12,7	13,5	0,8	15,2
$FX^{1}$	82,3	6,2	25,4	26,2	76,1	80,5	7,9	34,3	35,2	77,2	-1,8	1,7	8,9	9,0	1,1	9,2
	L*[- ]	a* [ - ]	•]*d	C*[- ]	[。] <b>u</b>	L*[- ]	a* [ - ]	b*[- ]	C*[- ]	[ 。] <b>u</b>	∆L*[ -]	Δa*[ -]	∆b*[ _ ]	∆C*[ -]	مه[ ]	ΔE*[ -]
Colour of native hardwood species			Intensifying colour, due wet surface			Colour difference										
	FA RO SO AL BT QU JG UL CP TI AC PY PR CT PO SA	FX <sup>10</sup> FA         RO         SO         AL         BT         QU         JG         UL         CP         TI         AC         PY         PR         CT         PO         SA           1 <sup>-</sup> 82.3         75.4         60.3         64.7         76.5         80.7         67.3         57.4         67.1         81.2         83.3         87.9         60.3         67.2         73.5         88.7         74.2	$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$	$  \  \  \  \  \  \  \  \  \  \  \  \  \$	$ \begin{array}{ c c c c c c c c c c c c c c c c c c c$			$  \   \  \  \  \  \  \  \  \  \  \  \  $	$  \                                  $			FY <sup>1</sup> FX         FX <th< th=""><th>FY<sup>1</sup>         FX         <th< th=""><th>Fx<sup>0</sup>         Fx         Ro         SO         AL         BT         QU         JG         UL         CP         PY         PK         CT         PO         SA           <math>\mathbf{I}^{-1}</math>         8.23         75.4         60.3         64.7         75.5         80.7         67.3         57.4         67.1         81.2         83.3         87.9         60.3         67.2         73.5         88.7         74.2           <math>\mathbf{P}^{+1}</math>         55.4         10.1         13.3         16.9         10.5         7.8         8.7         60.3         67.2         73.5         88.7         74.2           <math>\mathbf{P}^{+1}</math>         55.4         50.3         57.3         29.4         77.1         20.9         53.7         23.0         23.0         23.6         53.7         23.0         53.7         53.2         53.3         23.0         54.5         74.2         74.7         76.6         59.7         74.2         74.7         76.6         59.7         74.2         76.9         56.7         74.2         76.9         56.7         74.2         76.9         76.7         76.9         76.7         76.9         76.9         76.9         76.9         76.9         76.7         77.2</th></th<></th></th<>	FY <sup>1</sup> FX         FX <th< th=""><th>Fx<sup>0</sup>         Fx         Ro         SO         AL         BT         QU         JG         UL         CP         PY         PK         CT         PO         SA           <math>\mathbf{I}^{-1}</math>         8.23         75.4         60.3         64.7         75.5         80.7         67.3         57.4         67.1         81.2         83.3         87.9         60.3         67.2         73.5         88.7         74.2           <math>\mathbf{P}^{+1}</math>         55.4         10.1         13.3         16.9         10.5         7.8         8.7         60.3         67.2         73.5         88.7         74.2           <math>\mathbf{P}^{+1}</math>         55.4         50.3         57.3         29.4         77.1         20.9         53.7         23.0         23.0         23.6         53.7         23.0         53.7         53.2         53.3         23.0         54.5         74.2         74.7         76.6         59.7         74.2         74.7         76.6         59.7         74.2         76.9         56.7         74.2         76.9         56.7         74.2         76.9         76.7         76.9         76.7         76.9         76.9         76.9         76.9         76.9         76.7         77.2</th></th<>	Fx <sup>0</sup> Fx         Ro         SO         AL         BT         QU         JG         UL         CP         PY         PK         CT         PO         SA $\mathbf{I}^{-1}$ 8.23         75.4         60.3         64.7         75.5         80.7         67.3         57.4         67.1         81.2         83.3         87.9         60.3         67.2         73.5         88.7         74.2 $\mathbf{P}^{+1}$ 55.4         10.1         13.3         16.9         10.5         7.8         8.7         60.3         67.2         73.5         88.7         74.2 $\mathbf{P}^{+1}$ 55.4         50.3         57.3         29.4         77.1         20.9         53.7         23.0         23.0         23.6         53.7         23.0         53.7         53.2         53.3         23.0         54.5         74.2         74.7         76.6         59.7         74.2         74.7         76.6         59.7         74.2         76.9         56.7         74.2         76.9         56.7         74.2         76.9         76.7         76.9         76.7         76.9         76.9         76.9         76.9         76.9         76.7         77.2

 Table 1: L\*a\*b\* / L\*C\*h -colour values of native hardwood species and intensifying

 \_colour respectively colour differences (mean values of the specific sample set)

<sup>1)</sup> Abbr. according to ÖNORM B 3012

The colour difference  $\Delta E$  as a measure of the change of colour stimuli due to the wetting of the wood surfaces is shown in fig. 2.

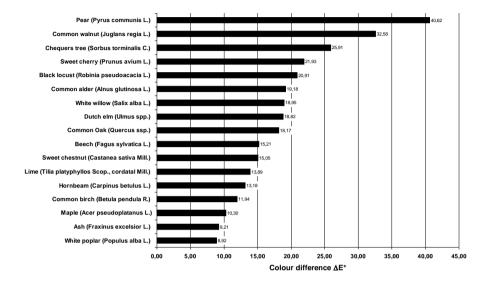


Figure 2: Colour difference  $\Delta E^*$  (indicator for accentuated colours) between colour of native hardwood species and intensifying colour, due to wet surface

#### CONCLUSIONS

For the most prominent domestic wood species a colour characterization could be provided based on the CIE-L\*a\*b\* colour designation system. The lightness L\* ranges between 57.4 (poplar, followed by maple) and 88.7 (walnut). The most intensive colour is represented by black locust whereas hornbeam is the most palish wood species.

Considering the whole spectrum of the colour space one can state that the colour range of the various domestic hardwood species tested is very limited and comprises 0.6% of the whole CIELAB colour space (fig. 3).

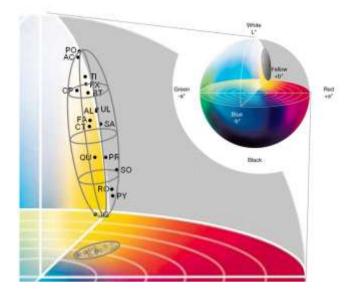


Figure 3: Colour of native hardwood species, shown in the L\*a\*b\* colour space

It could be shown how the various wood species react to wetting by a colour enhancement analyzed by the CIE-L\*a\*b\*-system. These data also support the understanding of the mechanisms of the colour enhancement as a physical phenomenon of light reflection and refraction on a "dry" and rough surface and a wet or clear coated glazed surface as discussed by Meichsner et al (2011).

Especially the huge variety of hardwood species benefits from the colour enhancement for a more intensive colour appearance which can be capitalized in the furniture and flooring industry. Understanding this colour enhancement also supports an innovative natural colour engineering for specific wood species.

#### REFERENCES

EN ISO 11 6664 Colorimetry - Part 4: CIE 1976 L a b Colour space (ISO 11664-4:2008)

FORSTHUBER, B., GRÜLL, G., TSCHERNE, F. (2010) Farblose Beschichtungen. Sonnenschutzmittel für Holz im Aussenbereich. Wiener Holzschutztage, Tagungsband. Holzforschung Austria, Wien, 105-111 FORSTHUBER, B., SCHALLER, C., GRÜLL, G. (2012) Evaluation of the photo stabilising efficiency of clear coatings compising organic UV absorbers and mineral UV screeners on wood surfaces. Wood Science and Technology, Online First, 26 June 2012

HON, D. N.-S., MINEMURA, N. (2001) Color and Discoloration. In: Hon, D.N.-S. and Shiraishi, N. (Eds.). Wood and Cellulosic Chemistry, Second edition. Marcel Dekker, Basel, New York, pp 385-442

HON DNS, SHIRAISHI N (2001) Wood and cellulosic chemistry, Chap. 9 Color and Discoloration, Chap. 11 Weathering and photochemistry of wood, Marcel Dekker, Inc, Basel, New York

LJULJKA, B. (1971) The effect of light on laquered wood surfaces. Holz als Roh- und Werkstoff, 29 (6): 224-231

MEICHSNER, G., HIESGEN, R., ESSLINGER, A., SCHOTTKA, A. (2011) Richtig anfeuern. Farbe und Lack, **117**(12), 24-28.

MITSUI K (2004) Changes in the properties of light irradiated wood with heat treatment: Part 2 Effect of light irradiation time and wavelength, Holz als Roh- und Werkstoff 62:23-30

MÜLLER U, RÄTZSCH M, SCHWANNINGER M, STEINER M, ZÖBL H (2003) Yellowing and IR-changes of spruce wood as a result of UV-irradiation, Photochem. Photobiol. B: Biology 69:97-105

NISHINO, Y., JANIN, G.R., CHANSON PIERRE, B., TIENNE, D., GRIL, J., THIBAUT, B. (1998) Colorimetry of wood specimens from French Guiana. *J* Wood Sci, 44, 3-8.

OLTEAN, L; TEISCHINGER, A; HANSMANN, C (2008) Wood surface discolouration due to simulated indoor sunlight exposure. Holz als Roh- und Werkstoff 66(1): 51-56

SULLIVAN, J.D. (1966) Color characterization of wood: Spectrophotometry and wood color. Forest Products Jorunal 17 (7): 43-48

TOLVAJ L, FAIX O (1995) Artificial ageing of wood monitored by DRIFT spectroscopy and CIEL\*a\*b\* color measurements, Holzforschung 49:397-404

TOLVAJ L, MITSUI K (2005) Light source dependence of the photodegradation of wood, Wood Sci. 51:468-473

URLAND, A. (1999) Colour specification and measurement. ARC Laboratory Handbook, ICCROM, Rome. ISBN 92-9077-157-7

WAGENFÜHR, R. (1996) Holzatlas. 4. neu bearbeitete Auflage, Fachbuchverlag Leipzig im Carl Hanser Verlag, München, Wien

# Microdensitometry of hardwood using helical X-ray tomography

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**Key words:** Helical X-ray tomography; microdensitometry; dendrochronology; vessel quantification

#### ABSTRACT

Nanowood is the latest multi-resolution X-ray tomography setup developed at UGCT, the Ghent University Centre for X-ray Tomography. It consists of an 8-axis motorized stage combined with two X-ray tubes and two X-ray detectors, specifically designed to obtain very high resolution scans as well as scans of larger objects. The system offers a large range of operation freedom, all combined in versatile acquisition routines (standard or fast scanning, tiling, helix, etc). It has a generic in-house developed CT scanner control software platform (Dierick *et al.* 2010) that allows full control of the scanner hardware. Reconstruction of the scans is performed with Octopus, a tomography reconstruction package for parallel and cone-beam geometry (Vlassenbroeck *et al.* 2007). The latest development includes GPU-based helix reconstruction, as such the scanner is suited for scanning elongated objects such as small beams and drill cores in order to obtain 3D information on the growth rings and density from pith to bark. For temperate hardwood species, 2D microdensitometry has already been used, yet its 3D equivalent has only recently been explored. Especially in the field of tropical dendrochronology, with often difficult growth ring demarcation, the concept of microdensitometry in 3D can possibly contribute to the study of growth cycles and the influence of the climate on wood formation. This approach can also assist in the demarcation of growth rings and visualization / quantification of wood anatomical traits such as vessels.

#### INTRODUCTION

X-ray tomography is a well-established technique and its multi-purpose use is widely recognized in wood science and technology. The number of papers with focus on lab-based X-ray tomography for fundamental research as well as its use as a tool for structural analysis is steadily increasing. A selection of different topics for which lab-based X-ray tomography can be used, shows the versatile employability in both static as dynamic experimental set-ups: wood anatomy (a.o. Van den Bulcke *et al.* 2008), structural wood vessel analysis (a.o. Brodersen *et al.* 2011), moisture dynamics (a.o. Lazarescu *et al.* 2010, Derome *et al.* 2011), analysis on wood derived materials (a.o. Faessel *et al.* 2005), etc. The non-destructive internal probing of a material at different scales, ranging from macroscopic down to microscopic level, is an enormous advantage. This paper elaborates on microdensitometry in 3D.

For temperate wood species, 2D microdensitometry has already been used, yet its 3D equivalent has only recently been explored. Especially in the field of tropical dendrochronology, with often difficult growth ring demarcation, the concept of microdensitometry in 3D can possibly contribute to the study of growth cycles and the influence of the climate on wood formation. This approach can also assist in the demarcation of growth rings and visualization / quantification of wood anatomical traits such as vessels. The technique enables to compile and archive dendrochronological series. microdensitometrical profiles and in some cases vessel chronologies. Examples on limba (*Terminalia superba*) and teak (*Tectona grandis*) illustrate the potential of the microdensitmetrical profiling while oak (Quercus spp.) is used to illustrate the potential of vessel quantification.

#### EXPERIMENTAL

The scanner used at Woodlab-UGent is a setup developed at UGCT, the Ghent University Centre for X-ray Tomography (www.ugct.ugent.be). It consists of an 8-axis motorized stage combined with two X-ray tubes and two X-ray detectors, specifically designed to obtain very high resolution scans as well as scans of larger objects (Figure 1). The system offers a large range of operation freedom, all combined in versatile acquisition routines (standard or fast scanning, tiling, helix, etc). It has a generic in-house developed CT scanner control software platform (Dierick *et al.* 2010) that allows full control of the scanner hardware. Reconstruction package for parallel, cone-beam and helical geometry (Vlassenbroeck *et al.* 2007). As the scanner set-up can be used in many fields of wood research, other experimental set-ups is referred to as well, such as the Continuous Moisture Measurement (CMM) set-up for non-destructive monitoring of the moisture content of wood and re-engineered wood materials (Van den Bulcke *et al.* 2009).



Figure 1: The multi-resolution X-ray tomography scanner Nanowood

# X-RAY TOMOGRAPHY SCANNING

Besides analyses deriving quantitative structural information from the volumes X-ray tomography scanning can be used to work with the data resulting from the underlying principle of a direct relation between greyscale and density. Although this relation is not straightforward and depending on the chemical composition as well, there is an opportunity of extracting densitometrical data from these volumes for wood as chemical composition in most cases can be considered more or less constant.

Since assessment of wood density variation and distribution can be studied very well starting from wood cores it is necessary to scan longer objects. Both reducing the magnification to capture the entire objects, which leads to reduced resolution, and performing multiple scans, which is cumbersome due to stitching the resulting volumes and also includes cone artefacts, are inadequate methods to perform high throughput analysis. To overcome this a fast and automated CT-scanning protocol was developed at UGCT for high-throughput 3D-analysis of microdensitometry on wood cores. Helix scanning is used complementary to cone beam scanning enabling gradient analysis over cross sections of trees (Van den Bulcke *et al.* 2012).

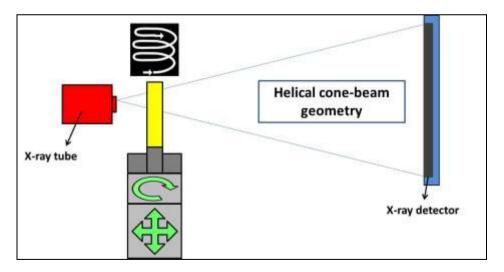


Figure 2: Helical cone-beam geometry implemented on the Nanowood CT scanner, a technical, mathematical and computational challenge.

The principle of 3D microdensitometry was explained and verified using helical X-ray tomography on wood cores in the paper by De Ridder and coworkers (2011). To obtain a resolution of 50  $\mu$ m, all cores are scanned with the Nanowood CT scanner using a closed microfocus X-ray tube reaching a spot size of approximately 30  $\mu$ m. Wood cores were mounted in a custommade holder made of a reference material with known density (Figure 3). This material was chosen as its atomic composition and density approach the composition and density of wood cell walls (1.56 g cm<sup>-3</sup>) (Kollmann 1951). By using this material and including the average grey level of air (zero density), grey values of reconstructed wood cores can be directly converted to densities.

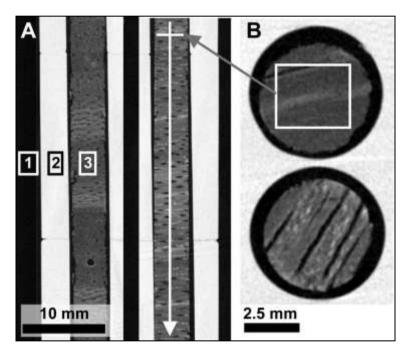


Figure. 3. A. X-ray scan of the reference material and wood cores: sectioning longitudinally through the holder (2) with 2 wood cores of limba(Terminalia superba) sectioned in different directions (3) and air (1) with indication of the travel direction of the frame used for average density calculation; b. Cross-section through 2 cores, with the rectangular frame shown as overlay on the upper core. (De Ridder et al. 2011)

Calibration of grey levels into densities was done by applying the following formula:

$$D_{i} = D_{holder} * (G_{i} - G_{air}) / (G_{holder} - G_{air})$$
(1)

where  $D_i$  = absolute density value of voxel i (g cm<sup>-3</sup>),  $D_{holder}$  is the absolute density value of the reference material (1.4 g cm<sup>-3</sup>),  $G_i$  is the grey level of voxel i,  $G_{air}$  is the grey level of air and  $G_{holder}$  is the grey level of the reference material.

Helical X-ray scanning reduces the time required to perform a full 3D-scan and volume reconstruction in comparison to classical tomography. The maximum length that could be scanned in one operation is now 16.3 cm. Therefore, some wood cores need to be split up so that the parts can be scanned separately. Broken wood cores were orientated correctly and recomposed with glue. Split or broken parts were stitched manually. After scanning and reconstruction, a rectangular window was placed in tangentially oriented slices (i.e. LT-slices, see Figure 3A and B) and the average grey level within this window was calculated. In addition, the average grey levels of air and of the reference material were also calculated. The average X-ray density of the wood within the window was then transformed to an absolute density value using equation 1. As such a 1Dmicrodensitometric profile based on a 3D X-ray scan of the wood core is obtained.

An example to illustrate potential is given in Figure 4 for limba (*Terminalia superba*) and Scots pine (*Pinus sylvestris*).

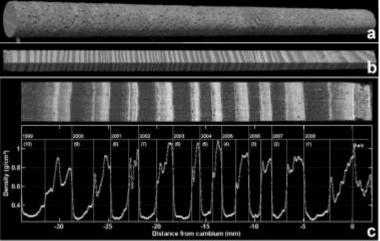


Figure 4: X-ray 3D volume of a core of limba (a) and of Scots pine (b) and microdensitometrical profile with ring width and age(c)

Although it is possible to analyse wood density microdistribution from 2D thin slides and identify mineral inclusion for e.g. oak as was illustrated by Vansteenkiste *et al.* (2007) in Figure 5, the 3D analysis allows for better interlinking with different wood tissues (vessels, parenchyma, fibres) and can be used for higher precision related to growth ring positioning and seasonal wood density patterns.

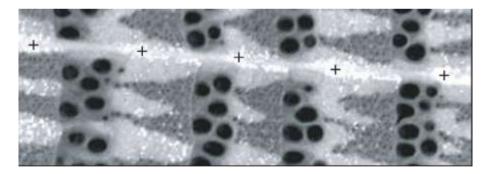


Figure 5: X-ray image of a short ring-sequence showing scattered and aggregated (+) pixels with (nearly) saturated grey-level (identified as mineral inclusions)s. Image size is 7.41 mm × 2.54 mm; scene extracted from a pith-to-bark strip of 0.7 mm thick. (Vansteenkiste et al. 2007)

Clearly 3D X-ray tomography allows separating the vessels in oak wood and so that ecophysiological impact of climate differences can be verified.

Number and size of vessels in oak (Figure 6) is a very adequate tool to study wood formation.

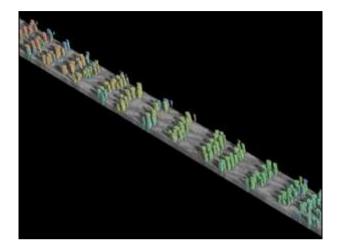


Figure 5: Distribution of vessels (number and size – diameter classes) by means of scanning a radial wood core (virtual strip).

Contrary to the regular structure of softwood, hardwoods in general and more specific tropical hardwoods might require additional corrections. Growth rings can deviate a lot from being parallel (non circular cross section, impact of roots – buttress roots). Furthermore grain angle can be considerable requiring additional corrections (spiral grain interlocked grain...). Recent work is focussing on these corrections as is illustrated by some scans of plantation Teak from Ivory Coast in Figure 7.

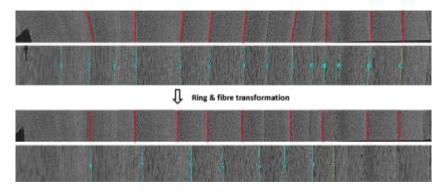


Figure 7: Growth ring and grain angle corrections needed for radial strip of plantation teak (Tectona grandis) from Ivory coast.

Based on fully corrected data set microdensitometrical variations can be used for determining growth ring borders and verify seasonal patterns. Although not an easy task when considering tropical hardwoods this tool allows studying hardwoods in far more detail with a reasonably high throughput. This potential is illustrated by a microdensitometric scan of afrormosia (*Pericopsis elata*) from DRCongo (Figure 8).

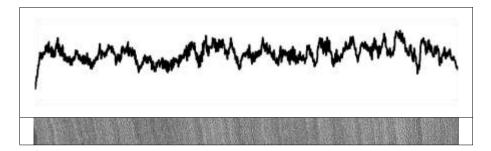


Figure 8: Seasonal variation in microdensitometry of afrormosia (Pericopsis elata) from DRCongo.

#### TRANSNATIONAL ACCESS VIA TREES4FUTURE

The Nanowood CT scanner equipment described and used in this paper is now also accessible for other research teams through the FP7 Trees4Future Research Infrastructures Project. More general information and specific information on the procedure for application can be found on the website (www.trees4future.eu).

The four-year Trees4Future project aims to integrate major forestry resources and infrastructures, to provide the European forest-based and wider research community with easy and comprehensive access to currently scattered sources of information and expertise. UGent-Woodlab gives access to the X-ray tomography scanner and the DSC-TGA equipment. The project provides a centralized access point via its web portal to major European databases in the area of forest genetics and forest ecology, and start to develop common protocols and reference standards for traits and species. Calls for transnational access are made, so that researchers can take advantage of the joint expertise, services and data of the 28 partners in the project. (*source:* http://www.efi.int)

### CONCLUSIONS

The enormous potential of flexible X-ray tomography scanners such as Nanowood can be used for fast 3D characterization of material but allows also for accurate microdensitometrical analysis of wood.

Together with other equipment for semi- to non-destructive testing, it offers wood research with the possibility to go a major step beyond current stateof-the-art. New scanning set-ups such as dual-energy and phase-contrast, and reconstruction algorithms such as the iterative family, will lead to faster scanning and faster and improved reconstruction and visualization of different, difficult to follow and discern phenomena and phases.

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#### REFERENCES

Brodersen, C.R., Lee E.F., Choat, B., Jansen, S., Phillips, R.J., Shackel K.A., McElrone, A.J. and Matthews, M.A. (2011). Automated analysis of threedimensional xylem networks using high-resolution computed tomography. *New Phytologist*, **191**(4), 1168-1179.

De Ridder, M., Van den Bulcke, J., Vansteenkiste, D., Van Loo, D., Dierick, M., Masschaele, B., De Witte, Y., Mannes, D., Lehmann, E., Beeckman, H., Van Hoorebeke, L. and Van Acker, J. (2011). High-resolution proxies for wood density variations in *Terminalia superba*. *Annals of Botany*, **107**(2), 293-302.

Derome, D., Griffa, M., Koebel, M. and Carmeliet, J. (2011). Hysteretic swelling of wood at cellular scale probed by phase-contrast X-ray tomography. *Journal of Structural Biology*, **173**(1), 180-190.

Dierick, M., Van Loo, D., Masschaele, B., Boone, M. and Van Hoorebeke, L. (2010). A LabVIEW® based generic CT scanner control software platform. *Journal of X-ray Science and Technology*, **18**(4), 451-461.

Faessel, M., Delisée, C., Bos, F. and Castéra, P. (2005). 3D Modelling of random cellulosic fibrous networks based on X-ray tomography and image analysis. *Composites Science and Technology*, **65**, 1931–1940.

Kollmann, F. (1951). Technologie des Holzes und der Holzwerkstoffe. Germany: Springer-Verlag

Lazarescu, C., Watanabe, K. and Avramidis, S. (2010). Density and moisture profile evolution during timber drying by CT scanning measurements. *Drying Technology*, **28**(4-6), 460-467.

Van den Bulcke, J., Masschaele, B., Dierick, M., Van Acker, J., Stevens, M. and Van Hoorebeke, L. (2008). Three-dimensional imaging and analysis of infested coated wood with X-ray submicron CT. *International Biodeterioration & Biodegradation*, **61**(3), 278-286.

Van den Bulcke, J., Van Acker, J. and De Smet, J. (2009a). An experimental set-up for real-time continuous moisture measurements of plywood exposed to outdoor climate. *Building and Environment*, **44**(12), 2368-2377.

Van den Bulcke, J., De Ridder, M., Dié, A., Toirambé, B., Vansteenkiste, D., Hubau, W., Van Loo, D., Dierick, M., Masschaele, B., De Witte, Y., Beeckman, H., Van Hoorebeke, L. and Van Acker, J. (2012). Microdensitometry of tropical wood using helical X-ray tomography: a proxy for dendroclimatology and forest management. In: *Proceedings of* 2012 IUFRO Conference on Division 5 Forest Products, p. 160.

Vansteenkiste, D., Van Acker, J., Stevens, M., Le Thiec, D. and Nepveu, G. (2007) Composition, distribution and supposed origin of mineral inclusions in sessile oak wood - consequences for microdensitometrical analysis. *Annals of Forest Science*, **64**, 11-19.

Vlassenbroeck, J., Dierick, M., Masschaele, B., Cnudde, V., Van Hoorebeke L. and Jacobs, P. (2007). Software tools for quantification of X-ray microtomography at the UGCT. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, **580**, 442-445.

# Aspects of beech wood (*Fagus sylvatica*) degradation after 7 years exposure in a modified L-joint test – a comparison between non-destructive and destructive evaluation

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**Keywords:** Beech wood, field testing, non-destructive, destructive evaluation, decay, discolouration, wood cracking.

#### ABSTRACT

The present paper is referring to some results of a modified L-Joint test, within which control and treated beech wood samples were exposed for 7 years outdoors, above ground. Different aspects of the complex degradation of wood and coatings after 7 years exposure, at macroscopic and microscopic level, are presented and the influence of a surface preservation treatment with a copper – chromium based reference biocide and finishing with two different coating materials is discussed. Non-destructive evaluation of the test samples (tenon and external faces) considered both biological and non-biological degradation of wood and was followed by a destructive evaluation. This consisted in sectioning the samples on radial longitudinal direction to reveal internal degradation. Discolouration and decay were noticed as result of progress from the tenon area or the surface of the samples towards the inner part of the samples. Wood cracking promoted or even caused internal decay and discolouration. The surface preservation treatment applied and/or coating had only a very limited protective effect. The variability of wood as a natural material corroborated with the reduced number of replicate test samples (3) and possible differences in the actual testing conditions (moisture content variations between samples) should be considered when analysing the experimental results.

# INTRODUCTION

Beech (*Fagus sylvatica* L.) is one of the most important hardwood species in Central and Eastern Europe with an area of about 12 million hectares. Beech forests account for approximately 19% of the forest area of Switzerland, 15% of Germany and 9.4% of the total area in France (PöHLER *ET AL*. 2006). In Romania about 31% of the total forests area is occupied by beech wood (http://dfwm.ugent.be/woodlab/docs/gottingen/cristescu.pdf) and approximately 4445 thousands m<sup>3</sup> of beech wood was harvested in 2010 (http://www.insse.ro/cms/files%5Cstatistici%5Ccomunicate%5Ccom\_anuale %5Csilvicultura/vol\_lemn2010r.pdf ).

The possibilities of improving beech wood properties, especially durability and dimensional stability for an extension and diversification of utilisation towards outdoors, above ground applications, corresponding to the use class (UC) 3 defined by EN 335, is a research topic of interest (http://www.erlauusa.com/Outdoor\_Furniture.html). The present approach cumulates novel technologies, such as acetylation or heat treatments with simpler technologies based on resins impregnation, alongside classical technologies based on preservation and surface coating. The efficiency of such technologies is often evaluated employing different standardised or original field-testing methods to obtain results easier to correlate to real utilisation situations (RABERG ET AL 2005).

The L-joint test, defined by EN 330 as standard method to evaluate the efficiency of wood preservatives to be used under a coating, represents one of the most employed methods to evaluate the initiation and progress of decay in wood exposed outdoors, above ground (CAREY AND BRAVERY 1986, DESPOT 1998, CLAUSEN AND LINDNEN 2011). The test samples mimic the bottom corner of a window frame with a poor quality joint, which facilitates water ingress and retention in the joint area, leading to fungal degradation. A modified, accelerated L-joint test was proposed by VAN ACKER and STEVENS (1997).

Within a long-time research project looking at the possibilities and limits of improving beech wood by simple surface treatments employing resins, biocide products and coatings, in order to improve its performance in outdoors, above ground exposure (UC3), a modified L-Joint test, adapted from the one proposed by VAN ACKER and STEVENS (1997), was employed to study the wood and coatings degradation phenomena occurring in time and a comprehensive evaluation scheme was developed for rating the occurring degradation phenomena (TIMAR *ET AL*. 2005, 2008, BELDEAN 2009). Some results after 5 years exposure were presented in the previous HWS conference in 2010 (TIMAR *ET AL*. 2010).

The present paper is referring to some results of this test after 7 years exposure. Non-destructive evaluation of the control untreated and treated samples was completed by a destructive evaluation after samples sectioning. Different aspects of the complex external and internal degradation of wood and coatings, at macroscopic and microscopic level, are presented and the influence of the surface treatments applied is briefly discussed.

#### **EXPERIMENTAL METHODS**

Beech wood (*Fagus sylvatica*) samples, representing the tenon members of L – joints (Fig. 1a), as those proposed by VAN ACKER AND STEVENS (1997), were investigated after 7 years of outdoors, above ground exposure. Details on the testing method and the comprehensive rating scheme were described in earlier publications of the authors (TIMAR *ET AL*. 2008, 2010).

After 7 years of exposure the non-destructive evaluation was followed by a destructive evaluation to reveal internal degradation of the test samples. For this purpose each sample was sectioned on longitudinal direction to result 3 slices as shown in Fig.1b. A first cutting was made through the actual tenon at 2mm from its lateral surface, whilst the second one was in the tenon shoulder, at 2mm from the other lateral surface of the actual tenon.

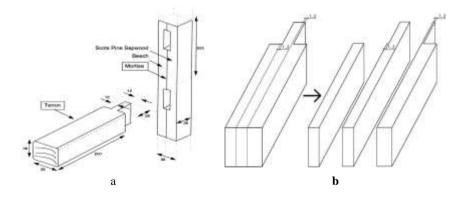


Fig.1. Methodological aspects of the modified L-joint test adapted from Van Acker and Stevens (1997): a. form and dimensions of test samples; b. sectioning scheme for destructive evaluation according SR EN 330

The test samples presented in this paper are: untreated controls, coded M, samples that were treated by short time immersion (15 minutes at 20°C) in a diluted aqueous solution (1%) of a reference Cu-Cr based preservative

(composition according to EN 330:  $CuSO_4$  5H<sub>2</sub>O - 50 %, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> – 48 %, CrO<sub>3</sub>- 2 %), coded R, and corresponding coated samples coded MS1, MS2 and RS1, RS2. These were prepared from control M and bio-protected R samples by finishing with two different coating materials: a semi-transparent, brown, alkyd lasure VILLA SUPRA (S1) and a white alkyd enamel SUPERPOLILAC (S2), made by Policolor (www.Policolor.ro). Coating was done by brushing in two successive layers on the external surfaces of the test samples. Three replicate samples were prepared and tested for each variant.

The non-destructive evaluation of the samples took into consideration both the inner joint area (tenon) and the whole external exposed surface (upper, lateral and lower faces), while destructive evaluation considered the whole area of the inner surfaces of the 3 slices (totally 4 faces). The rating scheme considered separately discolouration due to biological attack (C graded from 1 to 3 according to CEN/TS 12037:2003, Table C1), decay (D graded from 0 to 4 according to CEN/TS 12037:2003, Table C2), and cracks in wood (CW graded from 0 to 4 – original grading system). Macroscopic evaluation was completed by microscopic examination of the surfaces in reflected light using a stereomicroscope type Optika SZM 2 fitted with a PRO3 digital video-camera.

#### **RESULTS AND DISCUSSION**

#### Non - Destructive Qualitative Evaluation

The general aspect of the investigated test samples can be observed in Fig. 2, showing that a complex degradation of wood and coatings, including both biotic and non-biotic phenomena has occurred after 7 years of exposure and the differences in degradation of the non-directly exposed parts of the samples (the actual tenon of the joint) and the external, exposed surface of the samples. For the external surface the maximum and most complex degradation was registered on the upper face, the one directly exposed to sun and rain and receiving the maximum amount of UV radiation and water (coded a in Fig. 2). Less advanced degradation was registered for the lateral faces (coded b in Fig.2) and the lower faces (coded c in Fig.2), due to a reduced risk of wetting and limited UV exposure.

*Biological degradation* consisted in surface discolouration by stain or mould fungi and decay by rot fungi (*Basidiomycetes*), effects that appeared in time in this order, this being a common infestation sequence (DESPOT 1998, PFEFFER ET AL 2012 a, b).

*Discolouration* due to mould and staining fungi, mostly bluish-grey to black, was observed, at different intensity, for all the tested samples, no matter the treatment applied, being present both in the inner joint area (actual tenon) and on the external faces of the test samples. This was observed macroscopically (Fig.2) and microscopically (Fig. 3) as isolated spots or grouped in streaks or stains. For the uncoated samples the action of UV radiation and water also determined a colour change to grey making the rating of discolouration quite difficult, especially on the upper exposed face. For this reason microscopy was useful, as can be observed in Fig. 3a,b. Coating of wood with the two types of coating materials (S1, S2) only delayed initially biological discolouration, the formation of staining and mould fungi under the coating film and their further penetration through the coating was obvious for many samples and often the cause of the spot-wise degradation and/or micro-fissuring and further exfoliation of the paint film, especially for the alkyd white enamel S2 (Fig. 3 c,d,e,f)

*Decay in the joint area* (tenon) was the second form of biological degradation of the test samples (see Fig. 2 – details of tenons). For these samples the first signs of incipient decay as characteristic discolouration and/or wood softening (individual rates 1-2) appeared after 48 months of exposure (TIMAR *ET AL.* 2008). This type of degradation evolved after 7 years of exposure as severity (individual rates 1-3+, maximum average rate for a treating variant 2) and spreading, but was still not a general phenomenon.

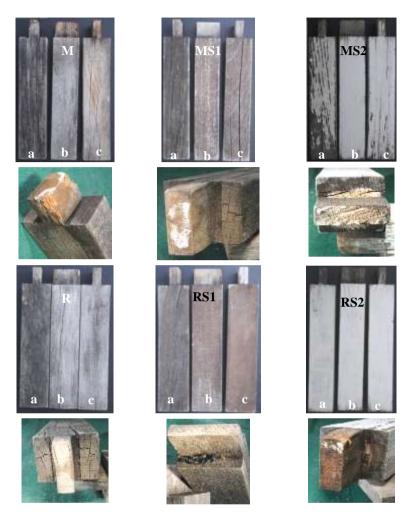
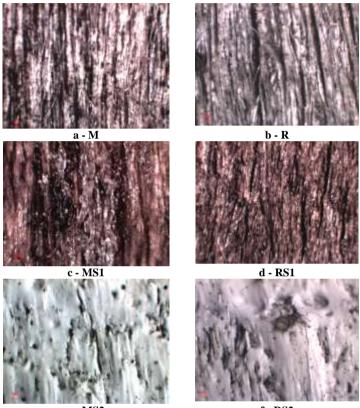


Fig.2. Beech wood (Fagus sylvatica) after 7 years outdoors, above ground exposure: general macroscopic aspect of the test samples on the three investigated faces (a- upper, directly exposed face; b- lateral face; c-lower face) and details of tenons degradation. (M - control untreated samples; R- samples treated with a Cu-Cr reference biocide; MS1, RS1 – samples coated with a semitransparent brown alkyd lasure; MS2, RS2 - samples coated with an opaque white alkyd enamel)

*Decay on the external faces* could be also observed for some samples, after 7 years exposure. However, only the upper faces of some test samples were affected, and even for the same batch of 3 replicate samples the occurrence of this type of degradation was different. It has to be remarked that at the previous examination after 5 years of exposure some incipient signs of

potential decay were noticed only exceptionally on the upper faces of a very few samples.



e - MS2 Beech – without bio protection

f - RS2 Beech – Bio protected

Fig.3. Microscopic features of external surfaces degradation (lower face) observed for uncoated (M, R) and coated samples (MS1, MS2, RS1, RS2) under a stereo-microscope at magnification of 40 x: a, b -discolouration spots, wood cracking, surface roughening, fibres detachment; c, d, e, f - micro fissures and cracks of the coating film, development of mould fungi under the coating film and penetration through the coating (bar 100µm)

*Wood cracking* was another major degradation phenomenon for both uncoated and coated samples. The high swelling and shrinking coefficients of beech wood and tensions associated to this dimensional variation in relation to the continuous climate change explain this form of degradation. Actually, the dimensional instability of beech wood counts more for its very limited exterior use than its low natural durability, which could be more easily corrected by adequate impregnation treatments with wood preservatives, considering the high treatability of beech wood (http://www.wolman.de/en/infocenter\_wood/from\_tree\_to\_wood/wood\_spec ies/beech/index.php). Furthermore, the extensive cracking in outdoors conditions will determine water ingress inside the wooden material and consequent decay.

#### Destructive Qualitative Evaluation - Internal Degradation of Samples

The non-destructive evaluation of the samples revealed biological degradation phenomena on the surface or starting from the surface and could only suggest sometimes a deeper extension of these towards the inner of the sample (decay).

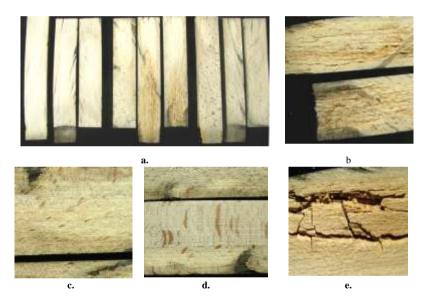


Fig. 4. Aspects of internal degradation of the L-joint test samples revealed after sectioning for destructive evaluation: a. general view of a batch of 3 replicates sectioned into 3 slices (M); b. detail of inner decay extended from the tenon area (M); c – detail of inner discolouration promoted from the surface / through the cracks (M); d. detail of inner incipient decay and discoloration around cracks (MS1); e. detail of extensive inner decay caused by extensive cracking (RS1)

The destructive evaluation performed after 7 years of exposure, by sectioning the samples, offered the possibility to actually see if and how the surface phenomena affected the inner area. Some aspects of internal degradation of selected samples are presented in Fig.4. Examining the pictures it can be seen that generally biological degradation evolved from the

surface towards the inside of the samples. For instance, decay which appeared in the tenon area developed along the grain within the sample affecting more than half of its length for an untreated beech specimen (Fig. 4 a, b).

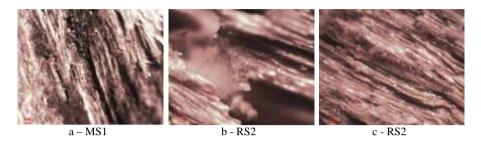


Fig.5. Microscopic features of inner biological degradation observed on sectioned samples under a stereo-microscope at magnification of 40 x: a) discoloration fungi, b) (white) rot fungal colonisation –mycelium in the proximity of a wood crack, c) fibre breaking in decayed area.

The same thing could be observed for discolouration by staining fungi, the phenomenon being more accentuated around the fissures and cracks in wood (Fig 4 c, d). Wood cracking, as result of repeated swelling and shrinking, not only promoted internal degradation by water ingress (Fig. 4 d), but also acted as the main reason for inner decay (Fig. 4 e). This proves the fact, the dimensional instability of beech wood and its extensive cracking in outdoors conditions is a key point to address in developing adequate treating technologies.

The internal biological degradation phenomena were examined also microscopically on areas of interest. The penetration and presence of discolouration fungi (Fig. 5a) as well as internal colonisation by (white) rot fungi (Fig. 5b) and wood structure disruption with fibre breaking due to decay (Fig. 5c) could be observed.

#### Non - Destructive vs. Destructive Evaluation - A Quantitative Approach

The graphs in Fig. 7, Fig. 8 compare the data resulted from non-destructive and destructive evaluation of discolouration and decay, while Fig.9 refers to wood cracking, a phenomenon promoting internal degradation. The degradation of the upper face of the external area and the tenon (inner joint area), which are the most susceptible to degradation, is compared to the internal degradation. The graphs are based on the mean values calculated for three replicates, but a high variability between the 3 replicate samples was noticed in some cases and, therefore, these results should be considered more

as a basis of discussion rather than a basis for final conclusions. More replicate specimens as well as a system of monitoring the actual level of moisture content in the different replicates would have been necessary for more uniform results, as shown by other authors (DESPOT 1998, CLAUSEN AND LINDNER 2011).

The data referring to the discolouration due to mould and staining fungi are cumulated in Fig. 7. For the external upper faces, the most susceptible to this type of degradation, the maximum rate of 3 was reached after 7 years exposure by most of the samples, indicating that the surface treatment of the test samples with the biocide product based on copper and chromium was not efficient for such a long period of time. Only slightly lower values of around 2.2 - 2.5 were obtained for the samples coated with the alkyd white enamel RS2 and MS2. Generally, discolouration of tenons was slightly lower compared to upper faces (rates 1.8 - 2.5), due to a slightly more difficult penetration of water. Discolouration due to staining fungi evolved slowly from the surface towards the inner of the samples, so that destructive evaluation after 7 years revealed internal discolouration especially in the areas near the external surfaces or in the proximity of cracks in wood, obviously at a lower rate compared to upper surfaces or tenon. The lowest rates (around 1.5) were registered for the samples treated with the biocide product (R. RS1, RS2).

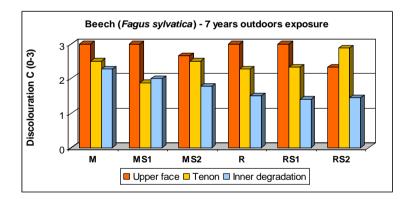


Fig.7. Influence of type of treatment on the discoloration of beech after 7 years exposure: upper faces, tenon and inner degradation (discolouration) of L-joint samples (M - control untreated samples; R- samples treated with the Cu-Cr reference biocide; MS1, RS1 – samples coated with the semitransparent brown alkyd lasure; MS2, RS2 - samples coated with the opaque white alkyd enamel)

The L-joint test is designed to promote water ingress and retention in the actual joint, so that the tenon is the part the most susceptible to decay of a

test sample. It is also expected that decay starting in the tenon area to progress inside the sample, most likely along the grain without being visible at the surface of the samples until quite late stages. The external joint areas, including the upper exposed face, are less susceptible to decay, because though they can get wet very quickly they will also dry more rapidly than inner joint area. The data referring to the non-destructive and destructive evaluation of decay, summarised in Fig.8, partly prove this expectation but also reveal other factors implied in the apparition and development of decay. One important factor is extensive cracking of wood which also promotes water ingress inside the wood leading to critical moisture content and delayed drying.

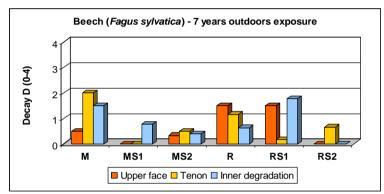


Fig.8. Influence of type of treatment on the decay of beech wood after 7 years exposure: upper faces, tenon and internal degradation (decay) of L-joint samples (M - control untreated samples, R- samples treated with the Cu-Cr reference biocide, MS1, RS1 – samples coated with the semitransparent, brown alkyd, lasure, MS2, RS2 - samples coated with the opaque white alkyd enamel)

For untreated beech samples (M), decay in the tenon, evaluated nondestructively, was rated at 2 and only incipient decay (rate 0.5) was observed on the upper face. Destructive evaluation revealed internal decay spread from the tenon area, rated 1.5. A quite similar situation, though at lower rates was observed for the coated samples MS2. In some cases (e.g. RS2) decay was registered only in the tenon area without any spreading towards inside the sample, whilst in other cases though no decay was observed during the non-destructive evaluation, internal decay was revealed by the destructive one (e.g. MS1). There were also situations (e.g. RS1) when decay started from the upper external face or the cross-cut end opposite the tenon due to extensive cracking and developed inside the sample, the tenon area being only little affected.

Regarding to the efficiency of the applied surface bio-protection treatment on the resistance to decay it is obvious that this could not impart resistance to decay which is a bulk phenomenon. Usually a vacuum impregnation treatment is recommended for wood application in UC 3 (e.g. windows), though even a treatment by dipping in an appropriate preservative before further coating should impart a better decay resistance compared to just coating, as shown by COOKSON (2010) on model windows after a test of 8 years exposure. However, the quantitative data from Fig.8 should be correlated to the qualitative aspects of internal degradation presented in Fig. 5 and the data from Fig. 9 referring to wood cracking, as decay apparition is mostly influenced by the moisture content in wood (RABERG ET AL 2005) and water ingress could be promoted by cracks in wood. On the other hand, degraded coatings could increase the risk of decay by allowing rainwater absorption into the wood and delaying wood drying, thus prolonging conditions suitable for fungal attack, whilst a bad fixture of the tenon in the mortise member could influence directly moisture content in tenon and risk of decay. Unfortunately, the actual moisture content within the samples could not be controlled during the test.

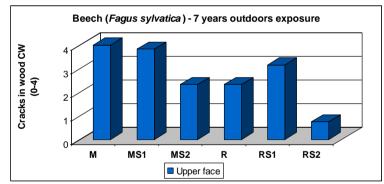


Fig.9 Influence of type of treatment on the non-biological degradation (cracks in wood) of upper faces tenon and inner degradation of the L-joint test samples after 7 years exposure: M- control samples without bio-protection, R- samples treated with the reference biocide

It can be noticed that most of the samples developed extensive cracking after 7 years of outdoors exposure, as expectable considering the dimensional instability of beech wood and the fact that for the test samples presented in this paper no treatment to reduce swelling and shrinking was applied. However, it could be noticed a positive effect of the treatment with the Cu-Cr biocide product in reducing the cracking of both uncoated and coated beech wood samples (R, RS1, RS2 compared to M, MS1, MS2). The complex reactions of copper fixation on wood under the influence of Cr<sup>VI</sup>

might have implied a modification of the hydrophilic character of wooden surface leading to a slower absorption of water in wood, as shown by MALDAS AND KAMDEN (1998) for CCA treated wood.

The samples with minimum cracking after 7 years of exposure were those bio-protected and further coated with the alkyd white enamel (RS2). The same samples presented the best behaviour in terms of external and internal biodegradation and the less degraded coating with a fairly good adherence of the coating film, though it is expectable that this situation will not have last much longer due to the mould fungi developing under the coating film and their negative effect on its adherence as illustrated by the microscopic pictures in Fig.3f.

#### CONCLUSIONS

Diversification and extension of beech wood (Fagus sylvatica) uses towards outdoors, above ground applications (UC3) is conditioned by adequate treatments to delay and reduce degradation. Studying and understanding the complex degradation phenomena occurring in time under these conditions and a realistic evaluation of the efficiency of the protective treatments employing appropriate field tests are key points to address.

A modified L-Joint test, within which control and treated beech wood samples were exposed and examined up to 7 years, was employed in this research. The treatments applied were exclusively simple surface treatments consisting in a preservative treatment with a reference biocide product and further coating with 2 coating materials.

Direct visual assessment and microscopy were employed to evaluate and rate the complex biological and non-biological degradation of beech wood on the inner joint area and external faces of the test samples. Non-destructive evaluation was followed by destructive evaluation by sectioning the test samples to reveal inner degradation.

The main degradation phenomena were discolouration by mould and staining fungi, decay in the tenon area and extensive wood cracking. Characteristic weathering effects were obvious for the uncoated samples, whilst the coated ones showed degradation of the coating films: spot wise degradation, cracking, adherence loss, flaking and exfoliation. The surface treatments applied had only a limited influence on the occurring degradation phenomena. The best results were obtained for the samples treated with biocide before finishing with alkyd white paint.

The destructive evaluation after 7 years of exposure offered a real insight into wood degradation by revealing inner decay and discolouration as result of progress from the tenon area or the surface of the samples towards the interior of the samples. Wood cracking promoted or even caused inner decay and discolouration.

The modified L-joint test applied in this research combined with the extended rating scheme developed proved their utility in monitoring the complex and interdependent degradation of wood and coatings in outdoors, above ground, long-term exposure.

### REFERENCES

BELDEAN, E. (2009). Dimensional Stabilisation of Wood - A Practical Issue of Topical Interest. In *PRO LIGNO*, vol 5(4):45-52.

CAREY, J.K., BRAVERY, A.F. (1986). Co-operative research project on Ljoint testing: Progress report to March 1986. IRG/WP/2272. *Inter. Res. Group on Wood Preservation*, Stockholm, Sweden. p.16.

CLAUSEN, C.A., LINDNER, D.L. (2011). Shading Aboveground L-joint and Lap-joint Tests: Comparison of White Pine and Sugar Maple Test Assemblies. *Forest Products Journal*, 61(3): 265-269.

COOKSON, L.J. (2010). Preservative treatments suitable for hardwood window joinery. *International Biodeterioration & Biodegradation*, <u>64 (7)</u>: 652-658.

DESPOT, R. (1998). Mechanism of infection of fir wood joinery; Part 2: Sequence and intensity of attack of micro organism, *Drvna Industrija (Wood Industry)*, 49 (3): 135-144.

MALDAS, D.C., KAMDEN, D.P. (1998). Surface characterization of chromated copper arsenate (CCA)-treated red maple, *Journal of Adhesion Science and Technology*, 12 (7): 763-772.

PFEFFER, A., MAI, C., MILITZ, H. (2012a). Weathering characteristics of wood treated with water glass, siloxane or DMDHEU. *Eur. J. Wood Prod.* 70:165–176.

PFEFFER, A., HOEGGER, P.J., KU, U., MILITZ, H. (2012)b. Fungal colonisation of outside weathered modified wood, *Wood Science and Technology*, 46: 63–72.

PÖHLER Evelyn, KLINGNER Raoul, KÜNNIGER Tina (2006). Beech (*Fagus sylvatica* L.) – Technological properties, adhesion behavior and color stability with and without coatings of the red heartwood, *Annals of Forest Science*, Volume 63, Number 2, p.129-137, ISSN (Electronic Edition): 1297-966X

RABERG U., EDLUND M.L., NASKO T., LAND C.J. (2005) Testing and evaluation of natural durability of wood in above ground conditions in Europe - an overview, *J. Wood Science*, 51: 429-440.

TIMAR, M.C., BELDEAN, E., MIHAI D. (2005). Opportunities and limits of wood improvement by surface treatment with reactive resins, *Proceedings of the Second European Conference on Wood Modification*, Goettingen, p. 178-185, ISBN 3-00-017207-6.

TIMAR, M.C., BELDEAN, E., ZELENIUC, O., LICA, D. (2008). Outcomes of a modified L-joint test in Romania In: Socio-economic perspectives of treated wood for the common European market, *Proceedings of COST E37 Final Conference*, Bordeaux, p. 107-117.

TIMAR, M.C., BELDEAN, E., LICA, D., (2010). Investigation of beech wood degradation alter long term exposure in outdoor, above ground conditions, *Proceedings of 4<sup>th</sup> Conference of Hardwood Research and Utilisation in Europe*, Sopron-Hungary, ISBN 978-963-9883-52-9.

VAN ACKER J, STEVENS M (1997). A new concept for the evaluation of wood durability for out of ground contact using accelerated L-joint testing, *IRG/WP 97-20123*, Stockholm, Sweden.

\*\*\* SR EN 335:2007. Durability of wood and wood- based products - Definition of use classes - Part 1.

\*\*\* SR EN 330:1997. Wood preservatives. Field test method for determining the relative protective effectiveness of a wood preservative for use under a coating and exposed out-of-ground contact. L-joint method.

\*\*\* CEN/TS 12037:2003. Wood preservatives. Field test method for determining the relative protective effectiveness of wood preservative exposed out of ground contact: Horizontal lap-joint method.

# Water absorption, Color Changes and Photostability of Benzoylated Beech wood Veneer

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Keywords: benzoylation, beech wood, artificial weathering, photostability.

#### ABSTRACT

Photodegradation of wood surfaces exposed outdoors is an important disadvantage of wood in service. Some physical properties as well as moisture absorption, weathering resistance and photostability of the wood can be improved by some chemical treatments. Also it has been proved benzoylation can improve the photostability of wood veneers. In this study, small wood specimens of beech wood (Fagus orientalis L.) veneers were esterified by benzoyl chloride. Wood veneers with 30 µm were cut from the radial face of beech sapwood. In this work, weight gain of samples after 3 and 6 hours reaction time, tensile strength before and after artificial weathering, water absorption and color changes and mass loss of veneers after modification and after artificial weathering were measured. Wood was modified to weight gain of about 92% after 3 hours. The increase in weight gain after 3 hours was insignificant compared to weight gain after 6 hours reaction time. Also the results showed that benzoylation resulted in a darkening of wood veneers. Tensile strength of treated samples was more than half of those of untreated samples. The results also indicated that tensile strength of modified and unmodified samples after 200 hours artificial weathering test was significantly lower than unweathered samples. Losses in mass of untreated and treated thin wood veneers after artificial weathering showed that benzovlation can significantly restrict the mass loss after weathering and UV irradiation. Measuring the color changes of samples after weathering presented highly better photostability of benzoylated beech wood veneers after 200 hours accelerated weathering tests. Results also indicated that 200 hours artificial weathering has caused the rapid changes in chromatic parameters specially lightness (L\*) and overall color changes  $(\Delta E^*)$  of unmodified veneers but not in modified samples.

## INTRODUCTION

Wood is one of the most important materials for many purposes in human life. Wood as a natural is susceptible to degradation by microorganisms, insects and fungi, also to change in dimension by humidity changes. It is very susceptible to surface photodegradation and weathering in outdoor conditions too. The main components of wood absorb ultraviolet radiation (Kalnins 1984). Absorption of UV photons can result in the formation of free radicals and that through the action of oxygen and water, a hydroperoxide is formed (Buerglund and Rowell 2005). Pandey and Chandrashekar (2006) reported the quantum energies collaborated with light are enough to break many of the chemical bonds present in wood components. Some solutions can be recommended to protect wood against photodiscoloration and photodegradation. The coating of wood surface can improve the wood surface quality exposed to UV radiation. The photodegradation of wood and other polymers can be controlled by UV absorber and additives too (Rabek 1990). Hence numerous commonly used wood and wood products must be protected by applying preservatives. Many traditional wood preservatives are harmful to human and animal's health because they contain trace elements or noxious compounds were

Several wood modification methods have developed in the last decades. One of the most important modification processes is chemical treatment. Most of the chemical modification methods have involved the chemical reaction of a reagent with the wood cell wall hydroxyl groups (Hill 2006). There have been many studies on the effect of acetic, propionic and butyric anhydride on wood. Etherification and esterification of wood and lignocellulosic materials are the chemical processes exchanging the ether or ester groups into hydroxyl group. Benzoyl chloride is a colorless liquid organochlorine compound and can react with wood. The extent of functional groups such as hydroxyl groups change in wood fibers is a result of benzoylation process which were determined by analytical techniques. It is found that Benzoyl chloride as a UV absorber can modify photostability of scots pine wood veneers (Evans 2002). Pandey and Chandrashekar (2006) investigated the photostability of pine wood esterified by benzovl chloride. They found this method was effective for the photostabilization of the wood polymers. Habu et al (2005) reported Sugi wood samples which were treated with benzoyl chloride exhibited higher resistance against brown rot decay.

In this study the effects of benzoyl chloride on beech wood photostability and color changes were carried out. The color changes before and after estherification by benzoyl chloride and after exposing UV were compared. Also color changes of wood samples were measured. Water absorption of beech wood veneers before and after treatment was investigated. Moreover the effect of benzoylation on the tensile strength of veneers was also investigated.

#### **EXPERIMENTAL METHODS**

#### Wood veneers modification

Beech wood veneers 50  $\mu$ m thick were cut from the radial surface of wood blocks with approximately 80 mm(longitudinal)×50 mm(tangential)×20 mm (radial) according to previously published method by Evans et al.(2002).

Before modification, veneers were conditioned at 65% relative humidity and 25°C for two weeks in laboratory. Digital micrometer was used to measuring the thickness of veneers.

Veneers were extracted with the mixture of acetone, ethanol and toluene (ratio 1:1:4) all purchased from Merck company, using a soxhelt extractor for 4 hours. Extracted wood veneers were oven dried at  $105\pm5$  °C overnight. Oven dried veneers were transferred to a desiccator over silica gel for 10 minutes and allowed to cool then weighted.

After extraction, samples were placed in polyethylene tubes. A mixture of benzoyl chloride and pyridine as a catalyst were added to tubes to completely immerse the wood. Benzoyl chloride was supplied from Merck Company. Tubes covered and placed in oil bath at 65°C for 3and 6 hours according to Evans et al (2002).

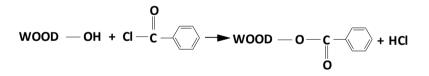


Fig.1. Scheme for the reaction between wood and benzoyl chloride.

After chemical reaction (Fig.1.), acetone was used to remove unreacted chemicals. After this step the specimens were oven dried at the 105°C and then weighted and weight percent gain due to chemical modification were determined. All control and modified samples were conditioned at 65% and 25°C for minimum 2 weeks.

#### Accelerated weathering test

Modified and unmodified Samples were subjected to artificial weathering test which was combined with UV-irradiation and water spray. Four replicates each of modified and unmodified wood samples were exposed to UV- irradiation for a total of 200 hours and water spray per 24 hour's cycles. Color coordinates L\*, a\*, b\*, tensile strength and water absorption of samples, before and after exposure, were measured. Also weight loss of specimens was measured before and after 50,100,150 and 200 hours artificial weathering.

#### Color determination

The changes in wood color before and after benzoylation and also after accelerated UV weathering tests were checked out by CIE L\*a\*b\* system. The color variation was recorded by Color Guide. In this study L\*a\*b\* values of the wood samples before and after modification with benzoyl chloride and after UV exposure were measured and data were used to determine the changes in color of samples. Total color changes  $\Delta E$  evaluate by the Commission International de l'Eclairage (CIE) L\*a\*b\* system from the changes in three spatial coordinates using the subsequent equation (Eq1.).

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$
(1)

Where color parameters  $L^*$ ,  $a^*$  and  $b^*$  respectively represent lightness (0)/darkness (100), redness (+)/greenness (-) and yellowness (+)/blueness (-) were obtained from measurements.

### Water absorption

Wood samples were placed into a beaker filled with distilled water. Weight of wood veneers had been recorded before and after soaking. Water uptake (WA) was calculated according to equation 2.

$$WA_t = [(W_2 - W_1)/W_1] \times 100$$
<sup>(2)</sup>

Where WA is the water absorption at time t,  $W_2$  is wet weight of wooden veneers after 4 hours soaking in water and  $W_1$  is initial oven dried weight of specimens.

## **RESULTS AND DISCUSSION**

#### Chemical changes after modification

Fourier transformed infra-red spectroscopy were acquired with the Thermo Nicolet Nexus 870 spectrometer. The analyses were carried out at room temperature and under an invariable relative humidity. Samples were analyzed using Kbr pellet. Fig.2 shows the FT-IR spectrum of control and modified veneers before and after weathering in the region between 500 and 4000 cm-1.

Wood veneers were reacted with benzoyl chloride satisfactorily, the appearance of strong absorption band at about 1727 cm-1 in benzoylated wood may be due to C=O banding. Other strong absorptions observed at about 1450 and 1055 cm-1. The infra-red spectra also show decrease in the intensity of the hydroxyl band at about 3436 cm-1.

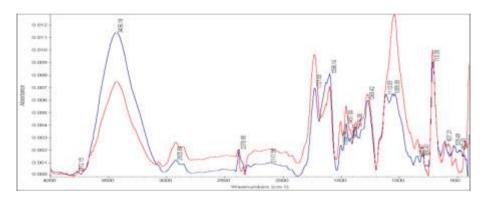


Fig.2. FTIR spectra of beech wood before and after benzoylation

### Weight percent gain (WPG)

After the extraction, WPGs of samples decreased significantly. Wood veneers obtained increase in WPG after benzoylation process. Average weight percent gain after 3 and 6 hours reaction were 89% and 92%. The high rate of chemical reaction can be explained by accessibility of thin veneer strips OH groups (active sites). The WPG increased with increment of reaction time insignificantly from the statistically point of view. Because of it, 3 hours reaction time was selected for this survey.

#### Water absorption before and after weathering

Water absorption of the unmodified and benzoylated veneers was about150% and 82% after 4 hours soaking respectively. The decrease in water absorption of modified veneers has been related to a decrease in the hydrophilic sites in wood such as hydroxyl groups. Water absorption of both of modified and unmodified specimens after artificial weathering was slightly increased. As expected the treated samples showed lower water absorption after weathering. After 200 hours artificial weathering water uptake of control and modified veneers were about 156% and 89% respectively. Chemical modification of wood is known to be an efficient method to obtain wood with high water repellency (Rowel 1983).

#### Color changes

Table1 shows the color changes parameters of unmodified and modified veneers before and after treatment. Libration of wood chromophoric carbonyl units after weathering can be related to photodegradation of the wood components specially lignin (Pandey and Chandrashekar 2006).

 Table1. Color coordinates of unmodified and modified beech wood before and after 200 hours artificial weathering

		L*	a*	b*
Before weathering	Unmodified	67.84 ±0.15	7.26 ±0.08	25.54 ±.07
	Modified	63.53 ±0.1	8.54 ±0.07	24.53 ±0.1
After 200 Hours Weathering	Unmodified	41.46 ±0.01	7.76 ±.06	23.46 ±0.04
	Modified	61.92 ±0.08	7.92 ±0.06	24.86 ±0.1

Whereas L\* and b\* after benzoylation were decreased slightly but a\* was slightly increased. There results showed that there was no noticeable changes observed in a\* and b\* after weathering in both modified and unmodified samples. Decrease in L\* value led to small darkening the color.  $\Delta E^*$  of control samples were 26.5 but in modified specimens were 8.6. These results indicated that artificial weathering to 200 hours has caused rapid changes in lightness (L\*) and overall color changes ( $\Delta E^*$ ) of unmodified veneers but not in modified samples.

### Weight loss after weathering

Artificial weathering had a large effect on the weight loss of veneers. The results here in accord with finding of Evans et al.(2002) and Chang and Chang (2001a and 2001b) suggest that esterification can reduce weight loss after weathering. Table 2 demonstrated the effect of benzoylation on the

weight loss after 200 hours accelerated weathering test at 50 hours intervals. The weight loss of unmodified and modified wood after 200 hours weathering was about 61.6% and 36.7% respectively.

Time of weathering (Hours)	Unmodified	Modified
50	42.1±2.9	22.5±1.1
100	48.8±8.5	25±0.1
150	53.4±4.8	30.9±0.1
200	61.6±3.6	36.7±1

 Table3. Weight loss of unmodified and modified beech wood before during artificial weathering

Weight loss of wood during weathering occurs because of photodegradation and leaching of lignin from the surface of wood (Derbyshire and Miller 198).

#### Tensile strength

Benzoylation reduced tensile strength of veneer strips. This result is in accordance with the findings of Evans et al. (2002). Table 3 shows the tensile strength of unmodified samples. Unmodified samples showed considerable losses in tensile strength after 200 hours artificial weathering. There are statistically significant differences between tensile strength of modified and unmodified veneers. Tensile strength reduction of unmodified veneers compared with modified ones after weathering were 44.3% and 33.9% respectively.

	Before weathering	After weathering
Unmodified veneer	25.3±6.2	14.1±0.8
Modified veneer	10.9±1.1	7.2±0.3

#### CONCLUSION

The results of this study suggested that benzoyl group can absorb UV irradiation. Benzoylation to high weight percent gain can play a useful role in improving photostability properties of beech wood veneers but it has negative effect on the tensile strength. It also can result in significant decrease of water absorption after modification. In general Benzoyl chloride is considered as a suitable modifying agent to improve performance of beech wood veneers. It can be explain by the effect of modification on lignin and cellulose.

#### REFERENCES

Berglund, L. & Rowell, R. (2005). *Handbook of Wood Chemistry and Wood Composites*. Boca Raton, FI: CRC Press.

Chang, H.T., and Chang, S.T. (2001b). Inhibition of the photodiscoloration of wood by butyrylation. *Holzforschung* 55, 255-259.

Chang, S.T., and Chang, H.T. (2001a). Comparisons of the photostability of esterified wood. *Polymer Degradation and tability*, 71,261–266.

Evans, P. D., Owen, N. L., Schmid, S., & Webster, R. D. (2002). Weathering and photostability of benzoylated wood. *Polymer Degradation and Stability*, 76, 291–303.

Evans, P. D., Wallis, F. A., & Owen, N. L. (2000). Weathering of chemically modified wood surfaces. Natural weathering of Scots pine acetylated to different weight gains. *Wood Science and Technology*, 34,151–165.

Habu N, Nagasawa Y, Samejima M, Nakanishi TM (2006) The effect of substituent distribution on the decay resistance of chemically modified wood. *International Biodeterioration & Biodegradation* 57, 57-62.

Hill, C. (2006). *Wood Modification. Chemical, Thermal and Other Processes.* Chichester: John Wiley & Sons.

Kalnins, M.A. (1984) Photochemical degradation of acetylated, methylated phenyl-hydrazinmodified and ACC-treated wood. *Journal of Applied Polymer Science*, 29(1), 105–115.

Pandey, KK & Chandrashekar, N. (2006). Photostability of wood surfaces esterified by benzoyl chloride. *Journal of Applied Polymer Science*, 99, 2367-2374.

Rabek, J. F. (1990) Photostabilization Of Polymers; Elsevier: London.

Rowell, R. M. (1983). Chemical modification of wood: A review, Commonwealth Forestry Bureau, Oxford, England, 6(12), 363–382.