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Nordic Forest Research  
Co-operation Committee (SNS)

Edited by  
Antanas Baltrušaitis  
Kristina Ukvalbergienė

# NORTHERN EUROPEAN NETWORK FOR WOOD SCIENCE AND ENGINEERING (WSE)

Proceedings of the 8<sup>th</sup> meeting  
September 13-14, 2012  
Kaunas, Lithuania

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**NORTHERN EUROPEAN NETWORK FOR WOOD  
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## **PREFACE**

Nordic Forest Research Cooperation Committee (SNS) in 2012 celebrated 40th anniversary of successful activities in promotion of research into the diverse functions of the forest resources in the biobased society. Jointed within the SNS-EFINORD cooperation networks aim to increase North European regional synergy within forest research by networking and joint utilization of unique research facilities. SNS supported Northern European Network for Wood Science and Engineering (WSE) was established in 2004 to promote research and increase competitiveness of wood and other forest products.

WSE network is continuously growing and in 2011 welcomed Germany and United Kingdom as participating countries. The field of the network covers wide aspects of wood science and engineering linking characteristics from macro to the sub-micro and molecular hierarchical levels. Traditional research areas include wood-water relations, wood durability, wood modification, wood mechanics, wood composites, engineered wood based products, timber structures, wood engineering and eco and energy-efficient use of machines and processes. The first seven meetings were organized by network partners in the different participating countries.

2005 – Norwegian Forest and Landscape Institute (Norway)

2006 –Royal Institute of Technology, KTH and Swedish National Testing and Research Institute, SP (Sweden)

2007 – University of Helsinki, Department of Forest Resource Management (Finland)

2008 – Latvian State Institute of Wood Chemistry (Latvia)

2009 – University of Copenhagen, Forest & Landscape Denmark (Denmark)

2010 – Tallinn University of Technology (Estonia)

2011 – Norwegian Forest and Landscape Institute (Norway)

The network proved to be efficient platform for the exchange of knowledge and facilitates better contacts between the forest research communities in the Nordic, Baltic Sea and the North Atlantic regions in the fields of wood science and engineering. Experienced and early-stage researchers including students have unique opportunity to meet, discuss, coordinate and encourage start-ups in cooperative state-of-the-art research and international projects. During the past seven years the network has constantly been growing, resulting in attracting 70 researchers from ten countries in 2012. The present proceedings contain 30 papers and 12 abstracts.

Department of Wood Technology at Kaunas University of Technology has the honor to host the meeting in 2012. We would like to thank all the authors for their contribution and the SNS for the financial support to make this event possible. The student award of 300 € this year is presented by our sponsor Boen Lithuania.

Antanas Baltrušaitis, Kaunas, September 2012



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# UNDERSTANDING PROPERTIES AND PERFORMANCE OF WOOD AT THE MOLECULAR LEVEL

Rowell, R.<sup>1</sup>

## INTRODUCTION

There are several levels of learning. The first level is our observations of some phenomenon. We may not understand what is going on but it has started our process of leaning. The next level is the accumulation of facts and data as we start to study the phenomenon we first observed. Finally, we start to understand what we have observed and studied. Even at the understanding level, there are three levels: the macro level, micro level and molecular level. The macro level is an understanding of what we observed, the micro is to understand the data from the tests we have conducted, and the final level of understanding is what is really going at the molecular level.

We can study the properties and performance of wood at all of these levels of learning and understanding. Table 1 shows the properties and performance of wood. Wood is hygroscopic and takes on moisture resulting in changes in dimensions; increasing in volume as moisture is sorbed and shrinks as moisture is lost. Because wood is an organic resource, it is attacked by a wide variety of micro and macro organisms. Wood is also degraded by ultraviolet radiation resulting in color changes and surface erosion. Wood is also degraded at high temperatures due to the production of volatile gasses and char at high temperatures. Finally, the strength of wood depends on the properties of the cell wall matrix.

**Table 1 – Properties and performance of wood**

<u>Element</u>	<u>Property</u>	<u>Performance</u>
Moisture	Hygroscopic	Dimensionally unstable
Decay	Organic	Microorganism attack
Ultraviolet energy	Phenolic	Color changes/surface degradation
Thermal	Unstable	Burns
Strength	High specific properties	Limited by matrix

Let us look at each of these properties at the macro, micro and molecular levels of understanding.

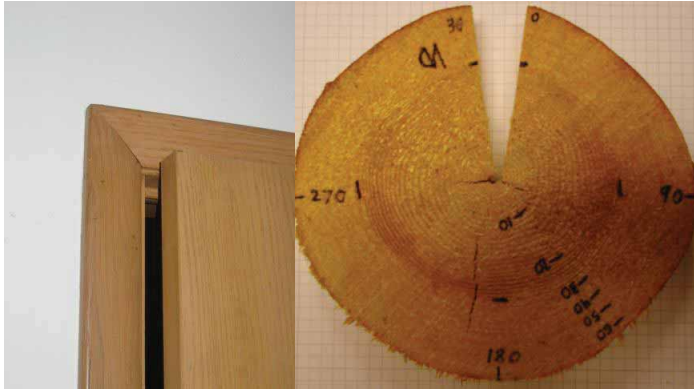
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<sup>1</sup> Professor Emeritus, University of Wisconsin, Madison, WI



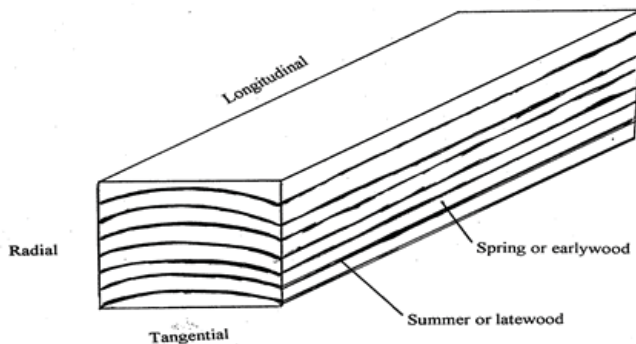
## MOISTURE

Our first level of wood and moisture is observing that our bathroom door has swollen in the spring and will not close. Or, we see that a freshly cut green log has split. The bathroom door has increased in size and the log has shrunk. We might even realize that the radial shrinkage is less than the tangential shrinkage in the log resulting in the “pie” type of split.



**Figure 1 – Observations that wood swells and shrinks as it takes on and loses moisture**

These first observations lead to a realization that wood is a non-isotropic resource meaning that it swells and shrinks to different extents in the three different growing directions of the tree. You may even run test to prove this or measure contact angle to see how fast a drop of water in sorbe into the wood.



**Figure 2 – The isotropic nature of wood: different in all three growing directions.**

Moisture is added to any hygroscopic surface in monomolecular layers and then building up layers of water after that (Figure 3). It is also possible to isolate each of the cell wall polymers and determine how they react with water (Figure 4). Now you see that the hemicelluloses sorb the most moisture.

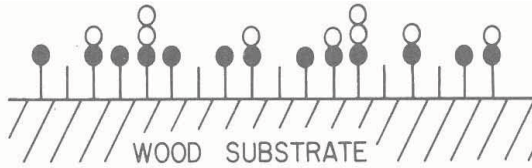


Figure 3 – Water added to wood in mono molecular layers.

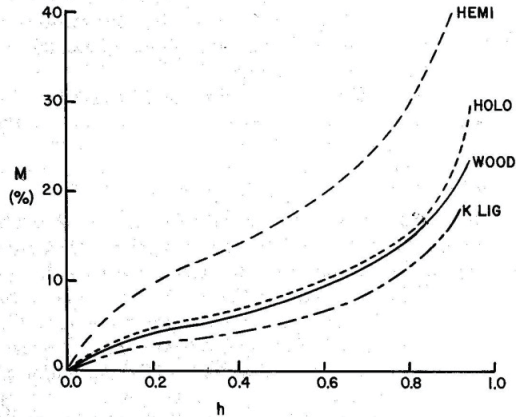


Figure 4 – Sorption isotherms for wood cell wall polymers and whole wood.

So, the polymer in the cell wall that sorbs the most moisture is the hemicelluloses that are located in the matrix of the wood cell wall (Figure 5). But, which of the hemicelluloses are mainly responsible for the sorption of moisture? A closer look at the structure of the hemicelluloses reveals that these polymers are all non-crystalline and contain side chains that might be even more accessible to moisture than the polymer chain (Figure 6).

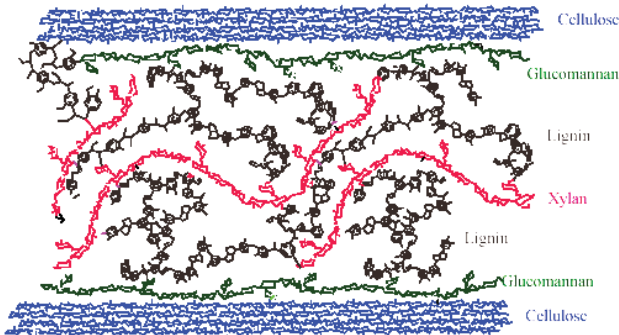
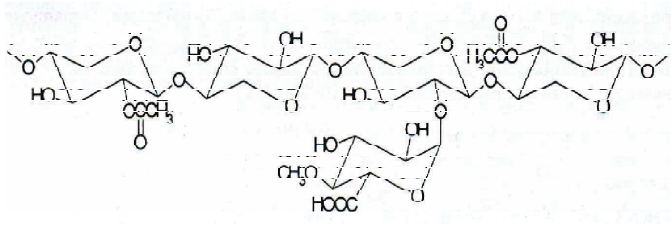


Figure 5 – Cross section of the wood cell wall.

Maybe it can be conclude that moisture sorption in wood takes place first in a side chain in the hemicellulose polymers at the molecular level.



**Figure 6 – Partial structure of a hemicellulose polymer.**

## DECAY

Your first observation that wood is degraded by micro and macro organisms may be a rotten log in the forest that has been attacked by fungi and termites (Figure 7).



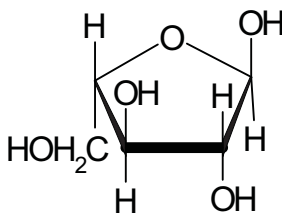
**Figure 7 – Log degraded by fungi and termites.**

You may decide to study this in the laboratory so you set up some standard tests to determine the types of organisms that attack wood (Figure 8). You will test different wood with different decay fungi and find that brown-rot fungi attack results in high weight and strength losses while white-rot fungi attack in a different way. But, why do the brown-rot fungi attack and what are they looking for? Since Nature does not waste energy, there must be a recognition mechanism that starts the decay process.



**Figure 8 – Tests for wood decay: upper left – ASTM standard soil block test, upper right – fungal cellar test, lower – in-ground stake test.**

Looking back at the structure of the cell wall (Figure 5) and the accessible side chains in the hemicelluloses (Figure 6), perhaps the first attack starts there? More specifically, maybe it is one sugar residue that is the first attack point. L-Arabinose is the only sugar in the hemicellulose structure that exists in a strained five membered ring (Figure 9). Is this the first recognition point at the molecular level for fungal attack to start?



**Figure 9 – Structure of  $\alpha$ -L-Arabinofuranose.**

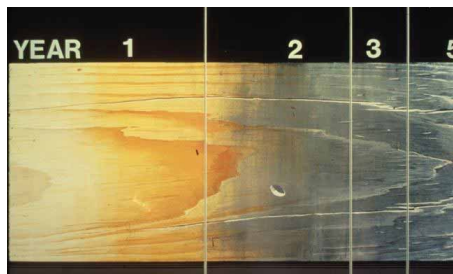
## ULTRAVIOLET ENERGY

Your first observation that wood is susceptible to degradation by ultraviolet energy may be the change in color of your newspaper or the roughness of the surface of an old wooden structure (Figure 10).



**Figure 10 – Change in color of news print and surface checking due to UV energy.**

You can run some standard tests on wood weathering in a weatherometer or outdoor tests and study the change in surface properties of wood and water due to ultraviolet energy (Figure 11). Your tests show that wood weathering is mainly a surface phenomenon and what is lost is mainly the carbohydrate polymers which are almost undegraded. What is degraded in the process is lignin which is the encrusting polymer in the cell wall (See Figure 5) hold the matrix together. Studying this process closer reveals



**Figure 11 – Changes in wood surfaces over time due to UV energy.**

that lignin undergoes an intermediate quinone-methide that is in a reversible tautomeric equilibrium with the free phenol (Figure 12). This starts the degradation of lignin at the molecular level.



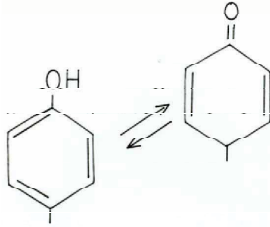


Figure 12 - Quinone-methide reversible tautomeric equilibrium with free phenol.

## THERMAL

We have all enjoyed a fire in the fire place or a campfire so we have observed that wood burns. Actually wood does not burn any more than the wax is burning when a candle “burns” (Figure 13). You study

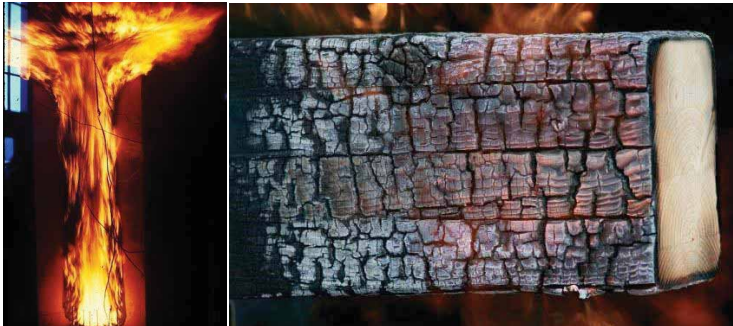


Figure 13 – Wood appearing to burn.

the thermal degradation of wood using several methods of analysis including thermogravimetric analysis and find that the hemicelluloses are the most susceptible to degradation at high temperatures (Figure 14).

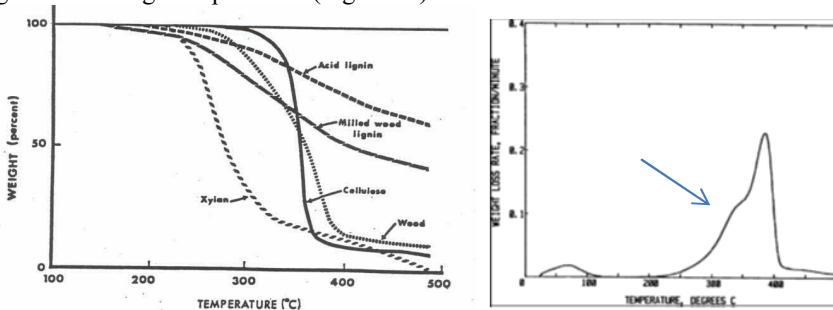
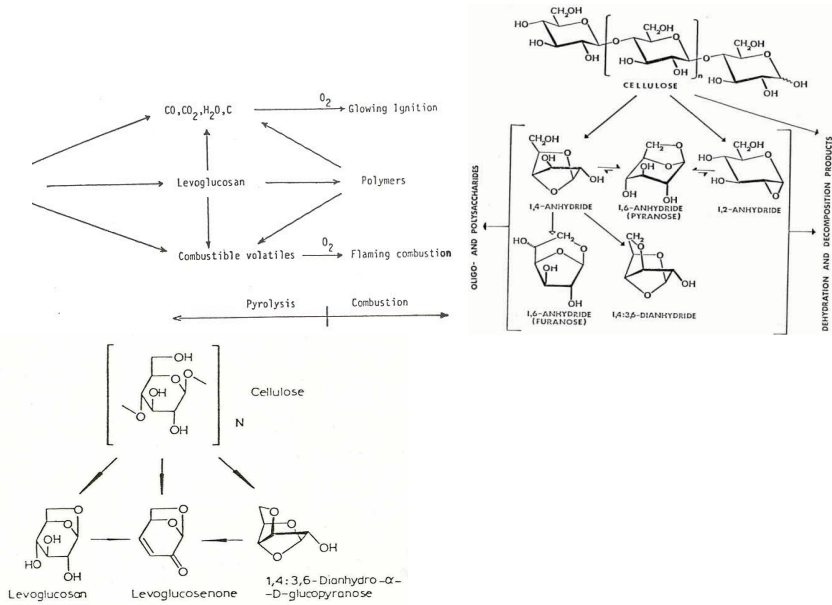


Figure 14 – Thermogravimetric analysis (TGA) of whole wood and cell wall polymers (left) and derivative thermogravimetric analysis (right) showing the hemicelluloses are the first to degrade (arrow).

Further study shows that flammable volatiles are produced during the thermal degradation of wood (Figure 15). Most of the isolated breakdown products derive from either cellulose or the hemicelluloses.



**Figure 15 – Mechanism of the thermal degradation of wood.**

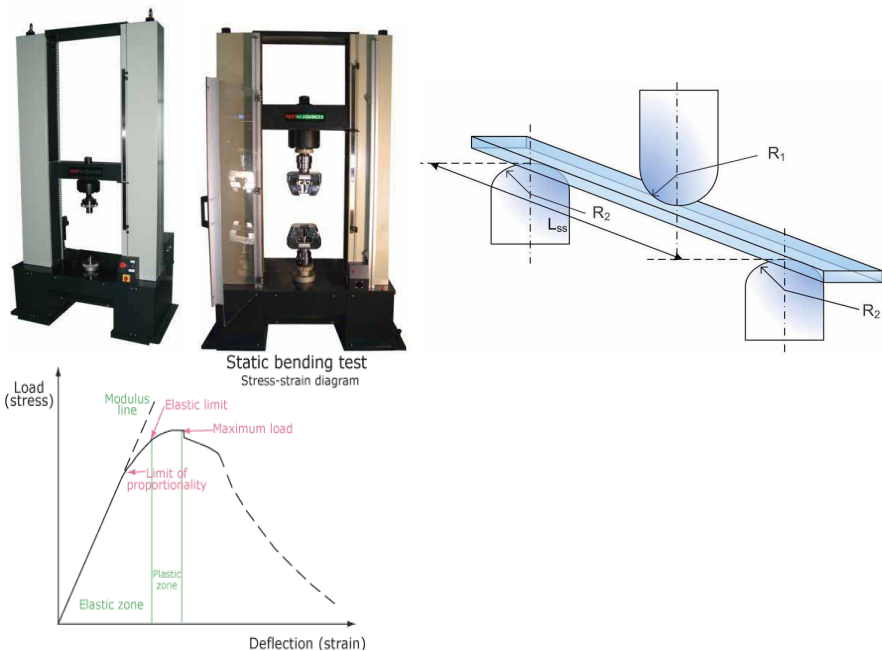
So, it can be concluded that the hemicelluloses are the key to thermal degradation at the molecular level.

## STRENGTH

The first observation of wood failure in a structure may be in a failed beam in a building (Figure 16). Obviously the wood has failed, and, in this case, in compression. Why did it happen? How did it happen? What failed? What is the C in crack and the B in break?



**Figure 16 – Wood failure in compression.**



**Figure 17 – Test machines to determine strength properties of wood and data from a bending test.**

We know that the specific tensile modulus of the cellulose micro crystal is 35 GPa but the specific tensile strength of whole clear wood is only 12 GPa. So, it is not the cellulose that breaks first. If it is not the cellulose that fails first, what does?

An analysis in the very early stages of strength loss in fungal attacked wood, there is a loss of several sugars in the hemicelluloses, namely, mannose, arabinose, galactose and xylose. This would indicate that the first failure occurs in the cell wall matrix between lignin, hemicelluloses and cellulose (See Figure 5).

So, strength is controlled at the molecular level by failures in the cell wall matrix.

## CONCLUSIONS

This paper has been a review of what we know and understand of wood properties and performance at several levels. Is it all true or do we have much more to learn?

# DECKING – SURFACE AND SYSTEM TREATMENTS AFTER TEN YEARS OF EXPOSURE

Alfredsen, G.<sup>1</sup>, Flæte, P.O.<sup>2</sup>, Schröder, A.<sup>3</sup>, Holte, S.<sup>4</sup> & Larnøy, E.<sup>5</sup>

## ABSTRACT

Wood for outdoor decking has a high market share in the Nordic and Baltic countries among private house owners. Important issues for the consumer are maintenance intervals and aesthetic appearance as well as decay resistance. Knowledge and consumer information about these aspects are required to ensure that wood can compete with alternative decking materials. In this paper an accelerated testing of decking, “stapelbädds metoden”, was evaluated after ten years of exposure at Ås, Norway. The test method covers different hazard situations within use class 3. Different preservatives and wood modification treatments were used in addition to untreated Scots pine (sapwood and heartwood) and larch (heartwood). The samples were treated with two different surface treatments. In addition there was one set without any surface treatment. Fungal discoloration and decay was evaluated. This provided new information about performance both on and above ground for a range of different combinations of preservative/modified systems and surface treatments of wood in decking for outdoor use. Generally, there were no significant differences in performance between the surface treatments, both with regard to surface discolouring fungi and decay fungi. For all surface treatments, the samples with rating 3 (heavy attack) in bottom layer in one or several stacks was: Tanalith M, Tanalith M (c), Gori Pres 10, Scanimp, styren, furfurylation, thermal modification, Ultrawood, larch heartwood, pine heartwood and pine sapwood. For all surface treatments, the samples with mean rating  $\leq 2$  (evident attack) in top and middle layer in one or several stacks was: ACQ 1900, Wolmanit CX 8, Tanalith E7, Gori SC 100, Royal, Royal with pigment, Scanimp, styrene and larch heartwood.

Key words: decay fungi, decking, discoloring fungi, use class 3

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## INTRODUCTION

In the Nordic and Baltic countries wood is a traditional and frequently used building material, also for outdoor decking. The service life of wood and wood-based products in outdoor applications is influenced by numerous factors, both wood-inherent properties (like extractives and species) and environmental factors (like climate, location, rain fall) (Nilsson and Edlund 1995, Brischke and Rapp 2008).

Product attributes that can influence the decision for purchasing outdoor wooden decking include environmental properties (i.e. silvicultural practices and chemical treatment), aesthetic properties, and tradition (Roos and Nyrud 2008) in addition to maintenance intervals and service life (both aesthetic and technical). Knowledge and consumer information about new wood protecting products are required to ensure that wooden decking can compete with alternative decking materials.

There is a range of European standards for laboratory and field tests to evaluate the protective effectiveness of wood preservatives both in soil contact (EN 252 1989) and above ground (EN 330 1993, ENV 12037 1996). There is some disagreement about whether or not the standard above ground test methods really are accelerated (e.g. Grinda et al. 2001, Grinda and Carey 2004). In the Nordic countries at least, they are time consuming because of the climatic conditions. Some of the new non-standard above ground tests like the block test (Pfeffer et al. 2008) and the double layer test (Rapp and Augusta 2004) use samples that are easy and inexpensive to prepare. They are accelerated using a shading device (for a more stable and humid climate) and/or feeder samples to speed up the decay. Another method is the “Stapelbädds method” (Edlund et al. 1997). The idea behind this method is that different moisture risk categories and types of biological attack are tested within the test block, combining on-ground conditions with two different exposure situations above ground.

The main objective of this study was to evaluate the performance of different combinations of preservative/modified systems and surface treatments of wood for outdoor use in decking, in order to give qualified consumer recommendations about different new products and systems.

## MATERIAL AND METHODS

In the project “Surface and system treatment of wood for outdoor use” the properties of different preservatives and wood modification treatments were tested with two different industrial surface treatment systems for above ground applications. The test started in 2002. This study reports data from Ås, Norway after ten years exposure. The test setup used was the “Stapelbädds method” according to Edlund et al. (1997), illustrated in Fig. 1. The wood samples had a dimension of 16 x 98 x 200 mm. Every test block contained ten samples with the same treatment, two samples in every layer, five layers in total. Each layer was orientated 90 degrees on the samples below. One block was used for each treatment. A fabric hindering weed growth, but permeable for moisture and micro-organisms, was used separating the bottom layer from direct soil



contact. Table 1 gives an overview of the treatments used. In addition, two different surface treatments: alkyd oil with iron oxide pigment (AO), and alkyd emulsions without pigment (AE) were tested in addition to no surface treatment (NO).

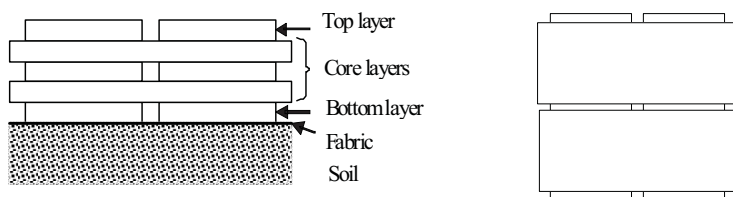


Figure 1. Illustration of the “Stapelbädds method” for on ground testing. The left picture illustrates the stack seen from the side, on the right the test seen from above. (Based on Edlund et al. 1997). Top layer: sample 1 and 2, middle layer: sample 3-8, bottom layer: sample 9 and 10.

Table 1. Wood species, treatments, abbreviations and active components used in the experiment. For each of these 17 treatments two different surface treatments: alkyd oil with iron oxide pigment (AE), and alkyd emulsions without pigment (AO) were tested in addition to no surface treatment (NO).

Wood species	Treatment	Abbreviations	Active component
<i>P. sylvestris</i>	ACQ 1900	ACQ	Cu, BAC
<i>P. sylvestris</i>	Wolmanit CX 8	Wol CX8	Cu-HDO, B
<i>P. sylvestris</i>	Tanalith E7	Tan E7	Cu, B, Triazole
<i>P. sylvestris</i>	Tanalith M	Tan M	Triazole
<i>P. sylvestris</i>	Tanalith M (with colour)	Tan M (c)	Triazole
<i>P. abies</i>	Gori SC 100	Gori SC 100	Triazole + IPBC
<i>P. sylvestris</i>	Gori Pres 10	Gori Pres 10	Triazole
<i>P. sylvestris</i>	Scanimp	Scanimp	Triazole
<i>P. sylvestris</i>	Royal*	Royal	Oil + Cu
<i>P. sylvestris</i>	Royal with pigment*	Royal (p)	Oil + Cu
<i>P. sylvestris</i>	Styren	Styren	Wood modification
<i>P. sylvestris</i>	Furfurylation	Furfuryl	Wood modification
<i>P. abies</i>	Thermal treatment	Thermal	Wood modification
<i>P. sylvestris</i>	UltraWood	UltraW	Water repellent
<i>L. decidua</i>	Heartwood	Larch heart	Untreated
<i>P. sylvestris</i>	Heartwood	Pine heart	Untreated
<i>P. sylvestris</i>	Control	Pine sap	Untreated

\* Only with no surface treatment.

The evaluation criteria were from 0-3. For discolouration: 0 = no discolouration, 1 = light discolouration, 2 = evident discolouration, 3 = total discolouration, for decay: 0 = no attack, 1 = light attack, 2 = evident attack, 3 = heavy attack. For every test sample the top surface and the bottom surface of each sample were evaluated separately.

## RESULTS AND DISCUSSION

After ten years all samples are fully covered of discolouring fungi except for bottom and/or middle layer of: Tanalith M with colour (NO and AO), Gori SC 100 (NO and

AO), Royal (NO), Tanalith M (AO), furfurylation (AE) and Ultrawood (AE) (data not shown).

Differences in decay between top and underside of boards, not differentiating between layers, are shown in Fig. 2. Generally, the decay pattern was the same for all surface treatments, and the top side had higher decay rating than the underside of the boards.

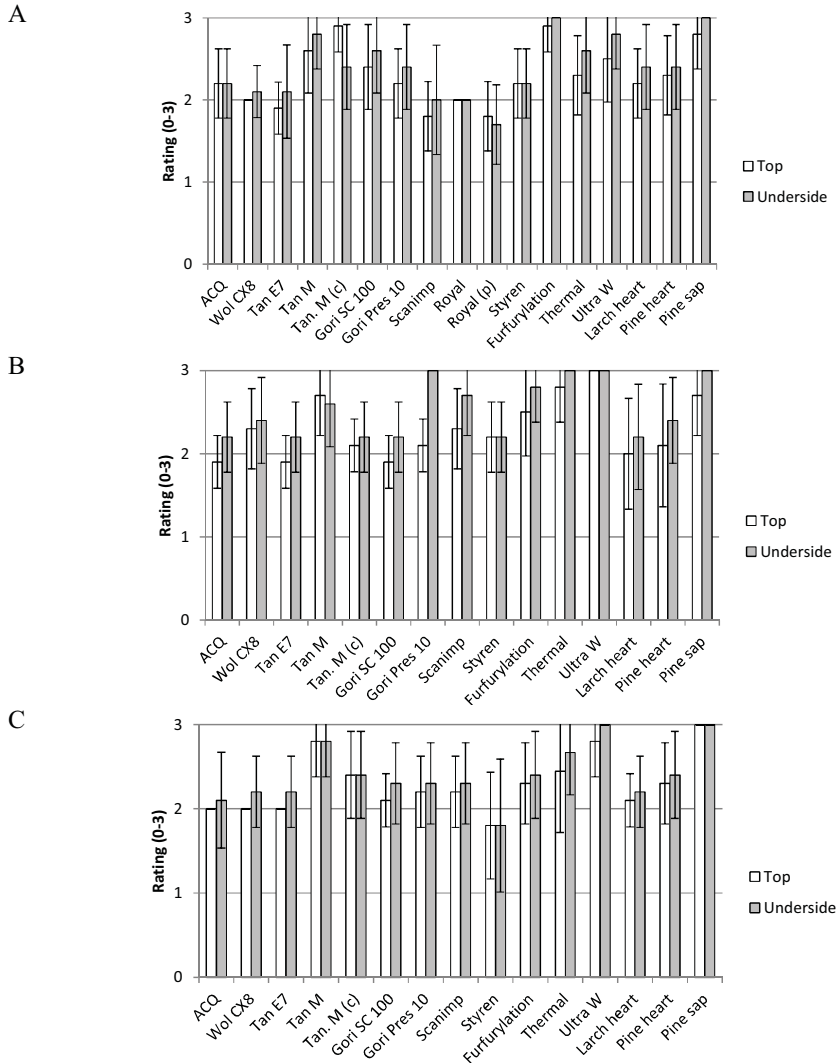


Figure 2. Decay top vs. underside of board – all layers included. A. No surface treatment (NO), B. alkyd oil with iron oxide pigment (AO), C. alkyd emulsions without pigment (AE).

A trend was that the bottom layer had the highest level of decay and the top level the least decay (Fig. 3). This was according to the expectations since the moisture exposure

is highest in soil contact. The study shows that the test method, at least to some extent, is able to imitate and differentiate between different moisture exposure situations. The treatments where the bottom layer had significantly higher decay than the two other layers were: Gori Pres 10, styrene, thermal modification, larch heartwood, pine heartwood (NO), styrene, larch heartwood, pine heartwood (AO) Tan M, Gori Pres 10, Scanimp, pine heart wood (AE).

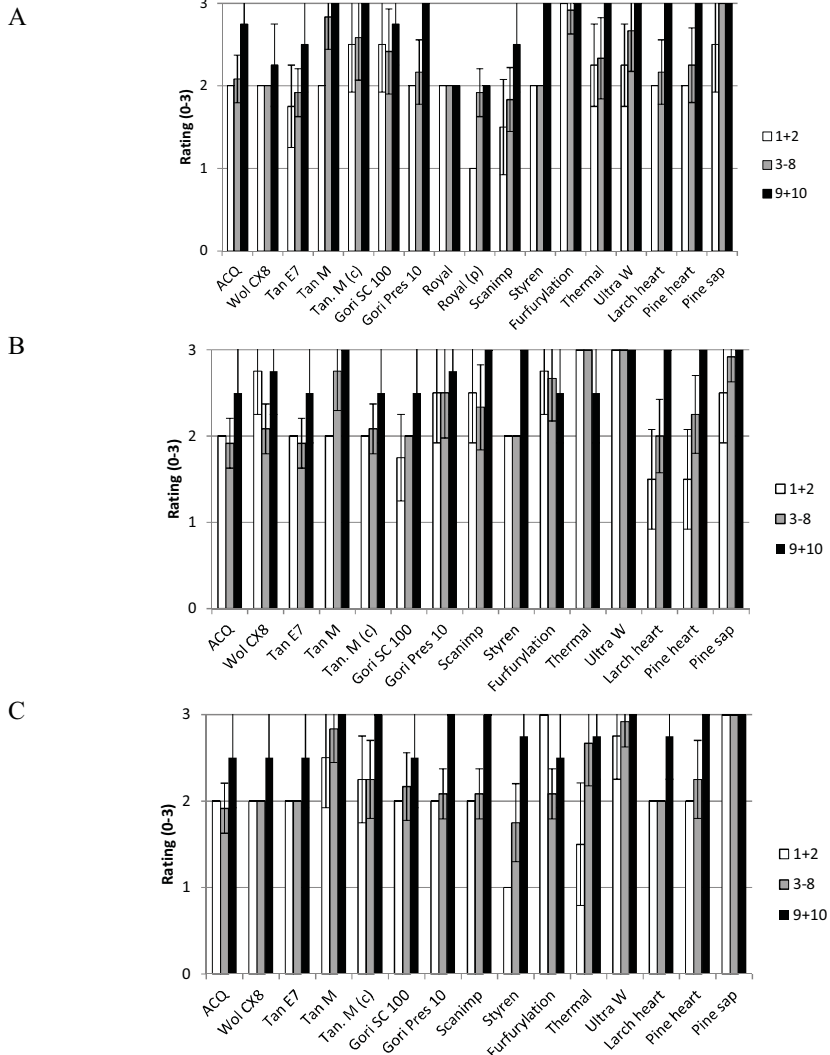


Figure 3. Decay between layers, top and underside evaluation of each board joined. A. No surface treatment (NO), B. alkyd oil with iron oxide pigment (AO), C. alkyd emulsions without pigment (AE). Top layer: sample 1 and 2, middle layer: sample 3-8, bottom layer: sample 9 and 10.

For all surface treatments, the samples with rating 3 (heavy attack) in bottom layer in one or several stacks was: Tanalith M, Tanalith M (c), Gori Pres 10, Scanimp, styren, furfurylation, thermal modification, Ultrawood, larch heartwood, pine heartwood and pine sapwood. For all surface treatments, the samples with mean rating  $\leq 2$  (evident attack) in top and middle layer in one or several stacks was: ACQ 1900, Wolmanit CX 8, Tanalith E7, Gori SC 100, Royal, Royal with pigment, Scanimp, styrene and larch heartwood.

The samples will be taken into the lab this autumn and analysed in more detail in collaboration with Georg-August-Universität Göttingen. Among the analyses planed are microscopy, chemical analyses of wood polymer content, fungal identification and quantification.

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# THE EFFECT OF NATURAL AND SYNTHETIC ANTIOXIDANTS AS A WOOD PROTECTIVE AGENT AGAINST WOOD DESTROYING FUNGI

Larnøy, E.<sup>1</sup> & Kolstad, S.<sup>2</sup>

## ABSTRACT

The use of wood in outdoor constructions has long traditions in Norway. In the last decade, the environmental demands for wood protective systems has become stricter. At the same time, new design solutions are arising that will challenge the decay protection in outdoor constructions. In this article, the potential for antioxidants as a wood protective agent was tested. Both natural and synthetic antioxidants have been used. All samples achieved a sufficient uptake of impregnation agent, and all the tested products showed a reduction in wood decay rate compared to the control. The protections by the antioxidants are more effective against brown rot fungi, than white rot fungi. The synthetic antioxidants stop the fungal degradation of the brown rot fungi, and should be considered as an additive in future and existing wood protective systems.

Key words: Antioxidants, *Coniophora puteana*, *Pinus sylvestries*, *Trametes versicolor*, wood protection.

## INTRODUCTION

The use of wood in buildings and constructions has long and good tradition in the Nordic countries. Wood is a heterogeneous and hygroscopic material. The building material from the Nordic wood species is generally not durable against biological degradation. Rot fungi influence the strength of the wooden material and mould and staining fungi causes discoloration. The degradation by brown rot fungi gives the most severe mechanical damages as it already at a low mass loss reduces the strength of the material dramatically. One way to protect weather exposed wood is to impregnate it with biocides. After the restrictions in the use of wood protection agents containing CCA (chromate, copper and arsenic), there has been an increased focus on finding new environmentally benign wood preservation agents.

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Oxidation is a substantial step in degradation process of brown rot fungi. Koenigs (1972, 1974) showed that brown rot fungi produce extracellular hydrogen peroxide  $H_2O_2$  and that wood contains iron ions, which lead to the hypotheses of that the Fenton reaction ( $Fe^{2+}+H_2O_2$ ) could be involved in the degradation of cellulose. The production of hydroxyl radicals from  $Fe^{2+}$  and  $H_2O_2$  from the Fenton reaction (Haber and Weiss 1934, Schmidt *et.al.* 1981) is showed as:  $Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + HO^*$  (Xu and Goodell 2001).

The produced hydroxyl radicals are assumed to be able to depolymerize long chained cellulose molecules to smaller fragments that easier can be taken up by the fungi. Due to this fact, several authors have tried to reduce the degradation of brown rot fungi by impregnating wood with commercialized synthetic antioxidants. (Schultz and Nicholas 2002, Goodell *et.al.* 2003). Antioxidants are molecules that protects against or reduce the oxidation of other chemical substances. An oxidation can cause damaging chain reactions leading to the degradation of cellulose. It is shown from earlier studies that Nordic berries contains high amounts of antioxidants, but the effect of antioxidants in Nordic berries against fungal degradation has not been addressed. The purpose of this test is to test the effect of natural and synthetic antioxidants against to wood degrading fungi's.

## MATERIAL AND METHODS

Samples of Scots pine (*Pinus sylvestries*) sapwood with the dimensions 30x10x5mm. The fungi tested were the brown rot fungi (*Coniophora puteana*) and the white rot fungi (*Trametes versicolor*). These two fungi were chosen as they are included in the European standard EN 113 (1996). The berries and impregnation solutions are presented in Table 1, and 25 samples per solution was impregnated. All the berries, except the elder, which was stored at 4° C, were stored at -20° C after harvesting.

Table 1. Impregnation solutions and antioxidant level of the solutions used in these trials

Impregnation solutions	Concentration	Antioxidantlevel(mmol/100g) (Halvorsen <i>et.al.</i> 2006)
Blueberries, <i>Vaccinium myrtillus</i>	100 %	8.23
Cowberries, <i>Vaccinium vitis-idaea</i>	100 %	5.03
Mountain ash, <i>Sorbus aucuparia</i>	100 %	2.42
Elder, <i>Sambucus nigra</i>	100 %	4.31
Black currant, <i>Ribes nigrum</i>	100 %	7.35
Ascorbic acid (pure vitamin C in the form of L-ascorbic acid (CAS nr. - 50-81-7) Sigma Aldrich)	17.6 % and 5 % (weight/volume)	
Propyl gallate (synthetic antioxidant >98 % (CAS nr. - 121-79-9) Sigma Aldrich)	1 % (weight/volume)	

The berries was first mashed in an OBH Nordica Blender, type 6658 and afterward juiced by a Philips HR 1861 juicer. The solutions was then stored at -20° C. The mass loss was calculated on the basis on the dry weight before start, dry weight after

impregnation and dry weight after the fungal trials on a scale with a resolution of 0.001 grams. The samples were dried at 103° C for 18 hours, and then acclimatized in an exsiccator before weighing. Afterwards, the samples were stored at room temperature. The samples were impregnated in plastic containers with a plastic net under and above the samples and weighted down. The impregnation scheme was 30 minutes of 40mbar followed by 90 minutes of 8 bars. The samples achieved different colour changes after impregnation (Figure 1).

The fungal degradation test was based upon the EN 113 (1996) test, and was performed with both test fungi. The samples were sterilized by 25 kGy Gamma radiation. Six replicates per treatment and fungi were used. Each petri dish contained one treated and one untreated sample (Figure 2). Additionally were to petri dishes with two treated samples per treatment with no fungal colonization for the calculation of the corrected value according to EN 113 (1996). For the test of fungal virulence, four petri dishes with to untreated samples were tested for both fungi.

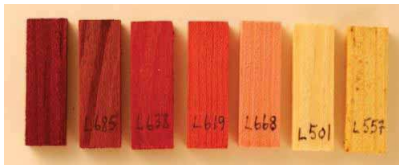


Figure 1. Colour change in the specimens due to impregnation of different solutions. From left: *Vaccinium myrtillus*, *Sambucus nigra*, *Ribes nigrum*, *Vaccinium vitis-idaea*, *Sorbus aucuparia*, ascorbic acid and propyl gallate. Photo: Sigrun Kolstad

The petri dishes used were 12 mm high with a malt / agar medium (4 % malt and 2 % agar). Autoclaved plastic net were used for sample support in the dishes. During the 8 weeks long test, the petri dishes were placed in a climatic room with 70 +/-5 % relative humidity (RH) and 20 +/- 2° C. The fungal mycelium was removed after fungal exposure.

Standard deviation and mean average were used in figure 3 and 4, and one-way ANOVA with Tukey HSD were used to compare means. Statistical software used was JMP 9.0 and MS Excel 2010.

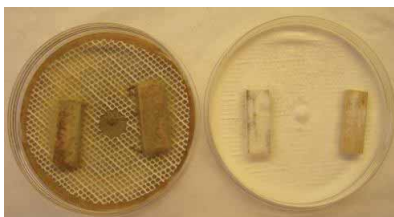


Figure 2. *Ribes nigrum* treated wood samples after eight weeks of exposure to the brown rot fungi *Coniophora puteana* to the left and the white rot fungi *Trametes versicolor* to the right. Photo: Sigrun Kolstad.

## RESULTS AND DISCUSSION

According to EN 113 (1996) should the virulence samples have a mean mass loss above 20% for brown rot fungi and above 15% for white rot fungi. In this test showed the two test fungi valid virulence with 28.8 % mass loss for brown rot fungi and 19.3 % mass loss for the white rot fungi.

All berry solutions showed a reducing effect on the decay by both fungal species with mass losses between 5 and 15 % (Figure 3). Brown rot fungi will normally give higher mass losses in Scots pine than white rot fungi. In spite of this fact, showed all treatments better protection against brown rot than of white rot fungi. This indicates that these solutions have a good effect against degradation by brown rot fungi. The lowest values were founded by *Vaccinium myrtillus* and *Sambucus nigra*. The untreated control samples shown al over a massloss between 25 and 35 % for brown rot fungi and 15 and 25 % for white rot fungi (Fig. 3).

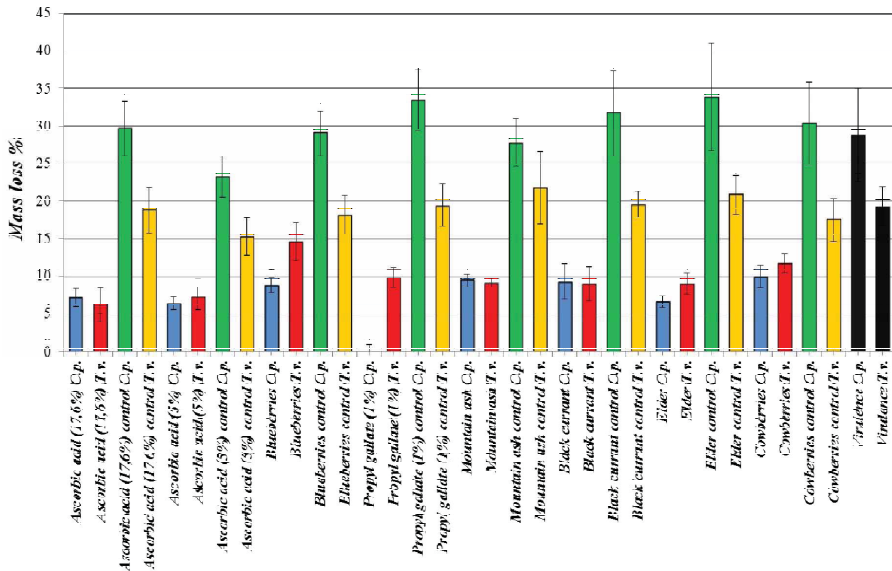


Figure 3. Different impregnation solutions tested against decay by brown and white rot fungi.

Ascorbic acid showed in both concentrations and for both fungal species significant mass loss reduction effect. By increasing the concentration of ascorbic acid from 5 to 17.6 % solution showed no significant decrease in mass loss. By further work with ascorbic acid should concentrations of 5 % or lower be used. The weight percent gain (WPG) was 25.9 % for the 17.6 % concentration and 8.1 % for the 5 % solution (Fig 4).

Propyl gallate (1 %) was the solution that provided the best protection against fungal degradation, and also the solution with the lowest WPG. The solution had a total inhibiting effect against attack by brown rot fungi and achieved a mass loss of 9.8 % when exposed to white rot fungi. For the further use of Propyl gallate against brown rot fungi, even lower solutions than 1 % should be considered. Propyl gallate should be considered as an additive in existing wood preservatives for an increased synergetic effect against fungal degradation.

Any further work with antioxidants should include a standardized leaching trial (EN 84 1997) for determination of the fixation effect.

In table 1 one can see the antioxidant level of the different berries on this trial. There were not found any correlation between antioxidant level and mass loss. Table 1 shows literature values and real content in the solution tested are not known.

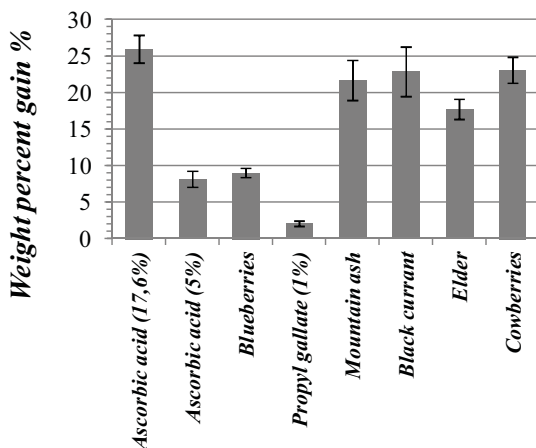


Figure 4. Increase in dry mass content after impregnation in percent.

## CONCLUSION

The results from this test showed that wood samples impregnated with natural occurring antioxidants in the form of pure berry juice achieve a higher anti-fungal effect than the controls.

Wood samples impregnated with propyl gallate and exposed to the brown rot fungi *Coniophora puteana* gave total protection. There were not found any correlation between literature antioxidant level in the berries and mass loss.

One commercial application from these results could be to add antioxidants to existing wood preservatives for enhanced synergetic effect against fungi degradation of wood.

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# EFFECT OF WOOD STRUCTURES ON THE OXIDATION OF UNSATURATED FATTY ACIDS

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## ABSTRACT

The aim of this study is to understand better the oxidation mechanism of the oxidation of fatty acids applied on wood surfaces. The drying process of air-drying fatty acid based systems has been extensively studied in coating systems but this has in most cases been performed on other substrates than wood. Currently, what is not understood is how wood components are chemically affected by the radicals involved in auto-oxidation and thus the effect of wood OH-radicals on unsaturated fatty acids has been evaluated in the present work. In the study presented here, the auto-oxidation process of methyl linoleate was measured in combination with wood compounds at 70 °C. The influence of wood model compounds, having phenolic, non-phenolic and polysaccharide structures, on auto-oxidation of methyl linoleate (ML) was observed by infrared spectroscopy (RT-IR). In our previous work it was observed that phenolic groups and radical conjugation are the main contributors to an antioxidant effect of lignin compounds on the oxidation rate of the methyl linoleate (Salehi 2010). The auto-oxidation process of methyl linoleate was measured in combination with 1 wt% reducing and non-reducing hemicellulose model compounds (HMC) at 70 °C. The effect of HMC on the methyl linoleate auto-oxidation process was also compared with the effects of glycerol and glyceraldehyde, using same analytical method and reaction conditions. The IR-spectra of methyl linoleate with 1wt% carbohydrates before and after oxidation were analyzed and peak intensity variations in the region of 3010 cm<sup>-1</sup> were calculated during the oxidation process. Hemicellulose model compounds were found to accelerate the radical reactions. Moreover, same analytical methods have been used to characterize the effect of different wood species on the mechanism of fatty

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acids auto-oxidation. The results indicated that hardwood have strong antioxidant activity on ML oxidation time, while ML oxidized faster with softwood (25 wt% relative to ML).

Key words: Auto-oxidation, Methyl linoleate, Wood model compounds, Softwood, Hardwood.

## INTRODUCTION

A common strategy for preventing outdoor wood degradation is to impregnate an anti-decomposition polymer or chemicals onto wood surfaces (Li 2011, Keskin 2011, Lesar 2011). Renewable materials such as fatty acids with hydrophobic properties on the porous wood surface can be used in an environmentally friendly system for outdoor wood protection (Lyon 2007, Ekeveld 2011). The coating process takes place by an oxidation mechanism. In addition, fatty acid auto-oxidation through radical formation in the drying process can be affected by different wood composites (Makowski 2005). In our previous study, the effects of lignin on oxidation of unsaturated fatty acids were analyzed by using infrared spectroscopy (RT-IR). In general, phenolic lignin or conjugated lignin structures were observed to inhibit the oil oxidation (drying) process (Salehi 2010). The aim of this work is to better understand the effects of different wood species on the auto-oxidation.

## MATERIAL AND METHODS

### Material

Methyl linoleate (ML) was used as oil model compound. ML (purity = 99%) was obtained from Sigma Chemical Co. (Sweden). Two different carbohydrates, hemicelluloses model compounds (HMC) representing different aspects of hemicellulose structure and wood model compounds such as lignin have been used in this study. All chemicals and ML are available commercially. In the present work both softwood and hardwood have also been used to analyze the effect of solid wood on the oxidation of fatty acids.

### Methods

For analytical purposes, hemicellulose model compounds (HMC) were each mixed separately with ML (1 wt% HMC relative to methyl linoleate) in 2 ml water and 8 ml ethanol to provide accurate measuring and homogeneous mixing. A typical recipe is 100 mg of HMC with 10 ml of water and ethanol in which 0.1 ml of this HMC-solvent solution was mixed with 99 mg ML.

To calculate the rate of the fatty acids oxidation with wood powder (25 wt% wood relative to methyl linoleate ML) different wood species were milled to 60-mesh particle

size. Real Time, RT-IR spectroscopy with ATR crystal-heater was used to oxidize the methyl linoleate mixed with wood model compounds or the milled wood. In order to observe oxidation of the samples by RT-IR, 10 mg of sample was placed on the open-air ATR crystal. During the Real Time-base Infrared (RT-IR) measurements, the heat stage was also set at 70 °C. Since the mechanism of oxidation of fatty acids may be readily monitored by RT-IR, both the initial oxidation product and final oxidation product of ML can be detected by RT-IR over time.

## RESULTS

The auto-oxidation of ML proceeds as previously described via a very complex reaction scheme where numerous reactions occur simultaneously (Porter Ned 1981, 1984, 1987). The initial phase normally starts with an induction period before the oxidation commences. The oxidation is then in the first stage dominated by a rearrangement of the double bonds from unconjugated *cis* double bonds to form a conjugated *trans* double bond system and a hydroperoxide. A second stage is then dominated by a consumption of conjugated double bonds to leave isolated *trans* double bonds in the system, which eventually will react. All this can be monitored using RT-IR following the peaks at 3010  $\text{cm}^{-1}$ , 990  $\text{cm}^{-1}$ , and 970  $\text{cm}^{-1}$  representing *cis*-unconjugated, conjugated *trans* and unconjugated *trans* double bonds respectively (Salehi 2010). The peak at 3010  $\text{cm}^{-1}$  is clearly seen in Figure 1, RT-IR spectra of pure ML at 70 °C before and after oxidation. The *cis*-unconjugated double bond was disappeared complete after 7 hours.

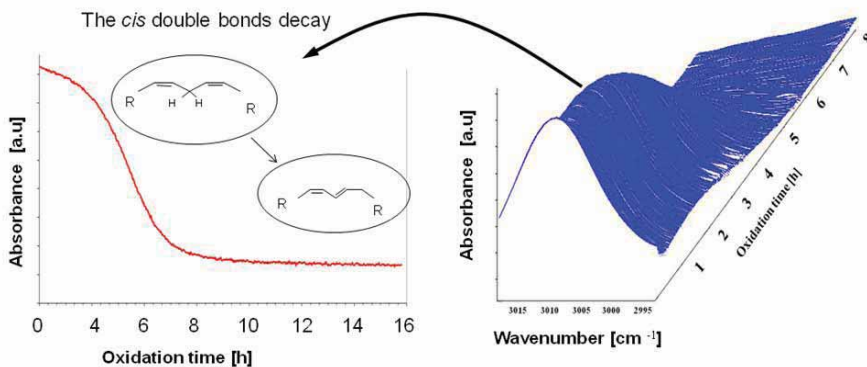


Figure 1: Fourier Transform Infrared (RT-IR) spectroscopy. Disappearance of *cis*-unconjugated double bond of pure methyl linoleate at 70 °C during oxidation time.

1 wt% aliphatic polyhydroxy compounds have shown that these compounds act as weak or strong oxidative effects on methyl linoleate. Lactose and sucrose caused a slight catalysis of the auto-oxidation of ML.

The RT-IR results showed that the degree of the oxidation rate observed by the effect on ML oxidation process with least to greatest effect being; wood model compounds < softwood < hardwood. However, The oxidation of ML with Kraft lignin (25 wt%) was not completed after 40 h.

## DISCUSSION

Since wood has a complex biological structure and has a lot of OH end groups both in lignin and cellulose, the exact position of radical interaction which affects the fatty acids during oxidation was not possible to identify by using RT-IR. However, the simple model compounds make it possible to understand in more detail about resonance stabilization of the radical. Most importantly, the results of oxidation method were obtained without using catalyzes addition in the system of ML-samples mixtures.

## CONCLUSIONS

Infrared spectroscopy is a powerful technique when aiming at the characterization of the changing of the functional group during oxidation process in real time. In the work presented here, we point out that the method for determining of fatty acids auto-oxidation affected by different wood species. The fatty acids oxidation were characterised by a variety of different wood model compounds and wood species. The results of this paper suggest that the lignin content and the wood structure may play an important role in the oil-wood interaction system during oxidation process. The results indicated that lignin inhibited the oxidation rate but hemicellulose compounds increased the oxidation time. Further, hardwood have stronger antioxidant activity than softwoods on ML oxidation time. A clear chemical interaction of fatty acids drying on different wood species can lead to improved wood protection for outdoor materials.

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# COMPARISON AND EVALUATION OF SEVERAL INDUSTRIAL AND EXPERIMENTAL COATINGS: EFFECTS OF ARTIFICIAL WEATHERING

Sansonetti, E.<sup>1</sup>, Kapaca, E.<sup>2</sup>, Cirule, D.<sup>3</sup>, Andersone, I.<sup>4</sup> & Andersons, B.<sup>5</sup>

## ABSTRACT

In this work, a set of solvent-borne coatings for wood protection and finishing, 7 industrial and 2 experimental, was studied to verify their performance and the effects of artificial weathering. The aim of this work was to evaluate the coatings' properties in terms of their hydrophobic properties, protection against UV, preservation of wood colour and formation of checks on the surface of samples. The wood used in this research was aspen (*Populus tremula*), which was thermally treated under the following conditions: 160°C for 3 h, and 170°C for 1 h, and also untreated specimens were used. Specimens were coated with 2 or 3 layers of coating, but for all of them the external layer was the same. Coatings were applied avoiding the formation of a visible film on the wood surface. Colour, surface integrity, and wettability were monitored before, during and after the weathering.

Key words: wood surface, coatings, artificial weathering, colour, wettability.

## INTRODUCTION

Both in outdoor and indoor use, wood needs to be protected against external agents, which degrade it and modify its aspect and chemical-physical properties. Humidity, light exposure, temperature, and biological attacks are factors that accelerate wood

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degradation. To preserve wood in good conditions, also esthetical, for a longer span of time, application of coatings and paints is still a widely used method. When exposed to severe environmental conditions, coatings lose their protective efficiency, so it is necessary to predict their in-service durability.

Use of laboratory methods, based on the combination of three parameters, namely, water, temperature and UV radiation, can help in predicting the durability of coatings with special devices that can simulate, in a shorter time, natural weathering effects. Accelerated ageing has been widely used in the coating industry over a long period of time, and many studies have focused on the optimisation of the involved parameters. Therefore, it is necessary to reproduce conditions similar to the real service conditions of wood, and there have been steady improvements, in the last decades, in this direction (Brunner et al. 2005, Perrin et al. 2001).

The choice of these parameters is important because different artificial tests differing in the UV light dose, sequence order of stress, and exposure duration can lead to different degradation phenomena, which could not correspond to the real ageing conditions of wood (Van den Bulcke et al. 2008). To evaluate the degradation of coatings, we focused the attention on the effects of artificial weathering, considering aesthetical and chemical-physical damages. In this research, of all the possible types, solvent-borne coatings were used, containing alkyd resins formulations. Of these, seven were commercial products, and two were prepared in the laboratory to compare their efficiency. These coatings were applied on samples of untreated and hydrothermally treated aspen wood to form three-layer and two-layer coated specimens.

## MATERIAL, METHODS AND RESULTS

### Coatings

Seven industrial coatings from different manufactures and two experimental coatings prepared in the laboratory were used in this experiment. A summary of the generic properties of the coatings is reported in Table 1, with the coating code, main component, aspect, solid content and density. All the coatings were solvent-borne and the solvent used for our experimental formulations was white spirit.

**Table 1.** Properties of the coatings

Coating code	Component	Aspect	Dry residual, %	Density, g/cm <sup>3</sup>
VC	Alkyd	Semi-transparent	20	0.80
Cp1	Alkyd	Semi-transparent	18	0.83
Cp2	Alkyd	Semi-transparent	6	0.81
Cp3	Alkyd	Semi-transparent	15	0.82
PTx	Alkyd	Transparent	5	0.80
TD	Alkyd	Transparent	24	0.83
Tk	Alkyd/linseed oil	Transparent	16	0.81
Exp1	Alkyd/linseed oil	Transparent	16	0.82
Exp2	Alkyd/linseed oil	Transparent	16	0.82

**Table 2.** Composition of coatings' layers

Coating system	layer 1	layer 2	layer 3
Tk2-VC	Tk	Tk	VC
Tk-VC2	Tk	VC	VC
Tk-VC	Tk	VC	
PTx2-VC	PTx	PTx	VC
PTx-VC2	PTx	VC	VC
PTx-VC	PTx	VC	
Cp1-VC	Cp1	VC	
Cp2-VC	Cp2	VC	
Cp3-VC	Cp3	VC	
TD-VC	TD	VC	
Exp1-VC	Exp1	VC	
Exp2-VC	Exp2	VC	

### Wood specimens

In this experiment, aspen (*Populus tremula*) wood was used. We prepared thirty nine specimens measuring 70 mm x 150 mm x 5 mm; they were planed to obtain a smooth surface. Thirteen specimens were untreated; thirteen specimens were thermally treated at 160°C for 3 h and thirteen at 170°C for 1 h. For each group, one specimen was used as the control without coatings and twelve were used to create three-layer or two-layer coated samples; each layer of the coating was applied with a brush in the tangential direction of wood grain, and allowed to penetrate and dry for 24 h. The amount of the wet coating applied on the wood specimens, as well as the weight of the sample during drying, was recorded using a balance with a precision of 0.01 g. To determine the dry residual of each coating, we placed 1 g of coating in an aluminium plate in an oven at 140°C for 1 h, and from this data the effective dry amount was calculated. Due to the differences in wood structure, pore dimensions and coating penetration, different specimens absorbed different amounts of coating: the applied wet coating varied from 62 g/m<sup>2</sup> to 180 g/m<sup>2</sup> and the dry residual varied from 11 g/m<sup>2</sup> to 33 g/m<sup>2</sup>. These values were calculated from the data reported in Table 1. The coatings were applied avoiding the formation of a visible film on the surface. The external layer was the same for all the specimens. After drying, wood samples were conditioned at 23°C and 63% RH for three weeks and then put in the weathering device.

### Artificial weathering

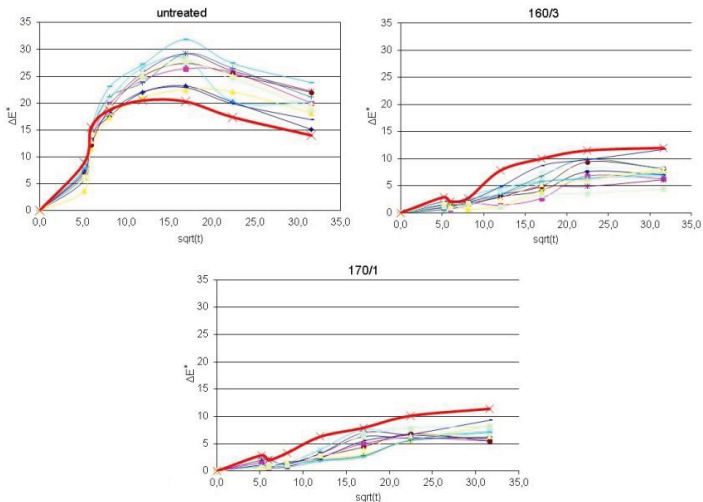
Specimens were weathered in a QUV Accelerated Weathering tester equipped with UVA-340 lamps, emitting in the wavelength region from 365 nm to 295 nm, with a peak emission at 340 nm. The test procedure was according to the EN 927-6 standard; one cycle consisted of 24 h condensation at 100% RH and 45 ± 3°C, followed by 2.5 h of UV irradiance at 60 ± 3°C and 0.5 h of water spray repeated 48 times. This cycle was repeated to reach a total of 1000 h, including 144 h of condensation, 715 h of UV irradiation and 141 h of water spray. At fixed intervals of time, changes in the specimens' colour and checks on the surface were recorded. Contact angle was measured before and after the complete weathering process.

## Colour measurement

Colour change is the most evident esthetical damage for exposed wood, and this concerns both untreated and heat treated wood to different extents. The main component responsible for wood discoloration is lignin, which strongly absorbs light radiation, initiating photochemical reactions that may ultimately lead to wood discoloration and photodegradation (Ayadi et al. 2003). The colour of the specimens was measured with a Konica Minolta spectrophotometer CM-2600d, equipped with a Xenon lamp. The colour changes are shown by three parameters, i.e.  $L^*$ ,  $a^*$  and  $b^*$ . The  $L^*$  axis represents the lightness, it varies from 100 (white) to 0 (black), and  $a^*$  and  $b^*$  are the chromaticity coordinates. Before, during and after the artificial weathering,  $L^*$ ,  $a^*$  and  $b^*$  colour coordinates of each sample were measured, based on a D65 light source by simulating the daylight. Fig. 1 shows  $\Delta E^*$  as a function of weathering time for all specimens, i.e. untreated specimens, and those heat treated at 160°C for 3 h and at 170°C for 1 h, respectively. Each value is a mean of ten points and each curve corresponds to one wood sample. For  $\Delta E^*$ , we have the following equation:

$$\Delta E^*_t = [(\Delta L^*_t)^2 + (\Delta a^*_t)^2 + (\Delta b^*_t)^2]^{1/2} \quad (1)$$

where  $\Delta L^*_t$ ,  $\Delta a^*_t$  and  $\Delta b^*_t$  are the colour coordinate changes.



**Fig. 1.** Colour changes during artificial weathering; the thickest lines are for uncoated specimens.

There is a different behaviour for untreated and heat treated specimens; untreated specimens show greater colour changes at the beginning, with a peak around 144 h of weathering for the uncoated sample, then the changes are lesser; coated specimens show an even greater change of colour, but this occurs later, after 288 h of weathering, so that the coatings seem to delay colour changes. This can be explained by a delayed leaching of photodegradation products from wood due to the presence of the coatings.

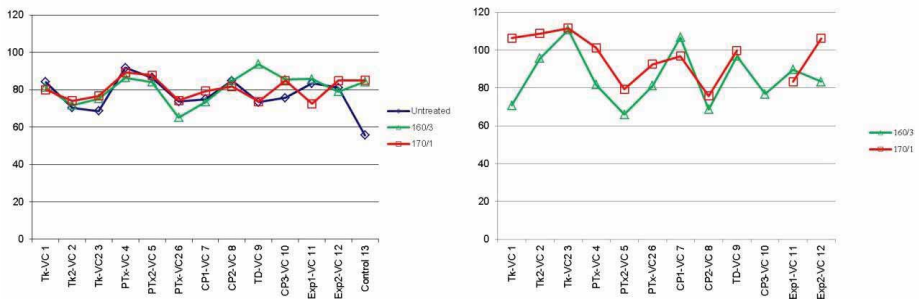


Heat treated wood shows a different tendency: coated specimens have lesser colour changes, they are more stable, but gradually growing during all the artificial weathering. Heat treated uncoated specimens show greater colour changes. This behaviour is probably a consequence of heat treatment, which causes lignin condensation, making wood more photoresistant (Ayadi et al. 2003).

### Contact angle

Before and after the artificial weathering, contact angle was measured. The goniometer used was a Dataphysics OCA20 device, equipped with a video camera and software for the determination and analysis of the drop contour, contact angle and solid surface free energy computation. Static sessile drop technique was used, which consists in the deposition of a single droplet on the solid surface without any further addition of liquid, and the liquid used was water: the device was equipped with an automated dispenser to have always the same volume of liquid, in this case 10  $\mu$ l. The results are listed in Fig. 2; the values are the average of six droplets for each sample.

Contact angles are reported before weathering for all the specimens, but after weathering are available only for heat treated specimens; untreated specimens, in fact, are strongly bended due to weathering, and the base line could not be correctly detected; hence, it was not possible to extract the entire drop contour, but it was observed that the damage on the surface made the drop to quickly penetrate inside the specimens, so that, in any case, it was not possible to obtain meaningful contact angle values or the contact angle was very small. Untreated wood showed a failure in coating efficiency if compared to heat treated wood; in this case, we have actually contact angles that, after weathering, are surprisingly larger, even more than 100°. It could be the consequence of the curing effect of UV radiations on coatings, at least at the first stages of weathering, but this must be still verified.



**Fig. 2.** Contact angle of specimens before (left) and after weathering (right). The Cp3-VC specimen value after weathering is not available due to the bending of the specimen.

### Formation of checks on the surface

Checks are the result of the swelling and shrinking of wood due to moisture changes during weathering, but also a consequence of the photodegradation of wood (Yata

2001). To prevent the surface checking of wood, coatings would need the incorporation of photostabilising compounds, for example, UV absorbers or reflectors. Surface damage was verified with an optical microscope, and the damage was evaluated with a scale from 1 to 5, where 1 means low surface checking and 5 means strong surface checking; controls were done after 144, 288, 504 and 1000 h. values are in Table 3.

**Table 3.** mean values of surface checking evaluation

time (h) sample	144 h	288 h	504 h	1000 h
untreated	3.1	3.6	4.0	4.6
160/1	1.9	2.8	3.7	4.2
170/3	1.6	2.5	3.7	4.3

On untreated specimens, checks form faster and are visible at lower magnification; heat treated specimens have smaller checks on their surface, but the final result is very similar: the formation of checks is slower due to their improved dimensional stability.

## CONCLUSIONS

The response of the coated wood to artificial weathering is different for untreated and heat treated specimens. In general, we can say that heat treated wood performed better than untreated one. It is not yet possible to eliminate the effects of external agents, but it is possible to improve the properties of coating to minimise those effects. On a short term basis, we know that good protection is obtained by using appropriate types and amounts of light stabilisers in primers and top coatings, combined with the stabilisation of the wood surface/coating interface (Butler et al. 2004). An important issue is also to find a performing combination of UV protectors and hydrophobic additives to reduce the checking of wood: traditionally, coating treatments have simply reduced checking by minimising moisture gradients and the magnitude of stresses that develop at wood surfaces (Evans et al. 2008). Surface water repellent treatments and wood preservatives that contain hydrophobes, like oils or waxes, work in this way and they are effective, but in a short term. Another important aspect is the coatings' transparency: transparent coatings have intrinsically more difficulty to block solar radiation, so underlying wood is subjected to photodegradation (George et al. 2005). Both untreated and heat treated wood shows damages from weathering, the presence of coatings helps to slow down degradation processes and leaching from the wood, but does not stop it. It is necessary to find other formulations and additives mainly to block the UV effects on wood.

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# NEW APPROACHES FOR STUDYING WOOD WETTABILITY AND LIQUID PENETRATION BY USING WILHELMY PLATE METHOD

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## ABSTRACT

Wettability studies on Scots pine veneers were done by using Wilhelmy plate method. The probe liquids were water and octane as swellable and non-swellable liquids, respectively. Novel approaches based on the Wilhelmy plate method to study wettability of wood veneers are introduced. Immersion to constant depth was performed and liquid uptake with time evaluated. Different kinetic regimes were observed, the fastest one associated with contact angle changes and the slowest regime associated with liquid sorption by capillary and diffusion. Two other approaches, imbibition at constant depth (with initial deeper immersion) and fully immersion, were developed in order to keep contact angle constant during measurements. Generally, water showed higher absorption than octane. In all wettability studies, and for both probe liquids, penetration processes started with a fast initial sorption due to filling the wood voids which followed by swelling in the case of water.

Key words: wood, Wilhelmy plate method, wettability, veneer, sorption

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## INTRODUCTION

Surface wettability plays an important role in wood science since wood is needed to be chemically modified, glued or coated in many applications. Many factors can affect wettability of wood surfaces including roughness, polarity, heterogeneity and porosity, wood grain direction (Shupe, Hse, Choong, & Groom, 1998), drying method (Wang, Zhang, & Xing, 2007). In addition, wood extractives can contaminate the probe liquids during test, change its surface tension and make errors on contact angle results (Nussbaum & Sterley, 2002; Wålinder & Johansson, 2001).

The most traditional and widely used method for measuring wettability of wood is sessile drop technique (Neumann & Good, 1979). But this method is valid only for homogeneous, flat surfaces at thermodynamic equilibrium. An immersion technique, Wilhelmy plate method, has been developed for measuring contact angle of porous materials such as wood (Gardner, Generalla, Gunnells, & Wolcott, 1991; Neumann & Good, 1979; Wålinder & Ström, 2001). The Wilhelmy equation for porous and hygroscopic material is:

$$F(h, t) = P\gamma\cos\theta + F_w(t) - \rho Ahg \quad (1)$$

where  $P$  is the wetted perimeter of the plate,  $\gamma$  is the surface tension of the probe liquid,  $\theta$  is the liquid-solid-air contact angle,  $\rho$  is the probe liquid density,  $A$  is the cross-sectional area of the plate,  $h$  is the immersion depth,  $g$  is the gravitational constant, and  $F_w(t)$  is the force due to wicking and sorption of the liquid at time  $t$ . If  $F$  (force) is plotted versus immersion depth ( $h$ ), the contact angle can be calculated. Dynamic sorption and penetration of liquids can thus be evaluated.  $F_A$  and  $F_R$  are obtained by linear regression of the advancing and receding curves to zero depth ( $h=0$ ), respectively. The intercepts are given by:

$$F_A = P\gamma\cos\theta_A \quad (2)$$

$$F_R = P\gamma\cos\theta_R + F_f \quad (3)$$

The Wilhelmy method solves the problem of heterogeneity by measuring the average contact angle over a larger area, the sessile method is still used as main technique to study wood surfaces (Ayrilmis, Dundar, & al., 2011; Lu & Wu, 2006; Mohammed-Ziegler, Hörvölgyi, Tóth, Forsling, & Holmgren, 2006).

The main purpose of the present study is to compare different methods for investigating wettability properties of wood veneers by using one swellable liquid (water) and one non-swellable liquid (octane).

## MATERIALS AND METHODS

The Scots Pine sapwood (*Pinus sylvestris* L.) dried at 104 °C for 3.5 h in oven, with dimensions of approximately (30 x 7 x 1) mm<sup>3</sup> (in longitudinal, radial, and tangential

direction, respectively) were used to study wettability of wood samples. First, pine lumbers were cut to smaller blocks with. Wood veneers were then prepared by cutting blocks along fiber direction using a wood chisel. Just after cutting the wood veneers.

Measurements were made using a Sigma 70 tensiometer from KSV Instruments. Ultrapure (Milli-Q) water (resistivity 18.2 M $\Omega$ ·cm) and synthetic grade of n-octane ( $\geq 99\%$ ) from Merck were probe liquids. Surface tension of contaminated octane (after wettability study with wood samples) also was measured and since no changes were recorded, octane was used for a series of measurements. Freshly cleaned glassware was used for measurements to minimize the effect of contamination of wettability study. Measurement conditions were approximately 22-23 °C and 35 $\pm$ 5 %RH.

### **Measuring dynamic contact angle**

Conditioned veneers were immersed in a small beaker (40 ml) filled with water. The velocity of 12 mm/min was used as optimum immersion velocity to minimize the wicking effects (Gardner et al., 1991). The veneers were vertically immersed to a depth of 10 mm and then withdrawn to 5 mm above the liquid surface. The contact angle was calculated from results of 0.5-2 mm depth of the first advancing curve. For measuring the actual perimeter of wood veneers, they were conditioned again to approximately same weight that they had before the first test. Thereafter they were immersed in octane which has contact angle zero with many types of wood surfaces. For perimeter determination the first receding curve of octane curve was used and then the perimeter of the veneers was calculated (Eq. 2.).

### **Imbibition at constant depth**

In this method, specimens were immersed rapidly to a depth of 5 mm and then rapidly withdrawn to 2 mm. The force detector measured weight gain of samples during experiment time. For this technique, the maximum velocity of instrument (44 mm/min) was applied for immersion and withdrawal stages. Octane measurement was done on reconditioned samples to calculate the real perimeter.

### **Immersion to constant depth (without withdrawal)**

The veneers were immersed rapidly to depth of 2 mm and the instrument recorded the force as a function of time. Water and octane were used as probe liquids, to study sorption in wood veneers. Same as previous method, the test velocity was the maximum for the immersion part. The specimens were conditioned to the same weight which they had before and the Wilhelmy plate method in octane was taken place to determine the actual perimeter of veneers. Finally the results of imbibition study were normalized by dividing by actual veneer perimeter.

## Full immersion

The veneers were horizontally immersed to depth of 9 mm in the small vessel filled with liquids. Since the width of veneers was about 7 mm, they had immersed completely in liquid. Liquid absorption was measured by measuring force in time. For this method, both edges of veneers were coated by polyurethane lacquer to protect against longitudinal penetration.

## RESULTS AND DISCUSSIONS

### Wilhelmy plate method-Dynamic contact angle

Fig. 1. shows a typical measurement from two cycles using the Wilhelmy plate method in water and octane to measure dynamic contact angle. A major feature of the water curve is the unevenness and curvilinear shape of the first advancing curve which does not appear in octane curve. This indicates that the surface of wood is heterogeneous and that each point of it has a different contact angle from its surrounding points. This is one main reason that this method has been recently preferred over sessile drop method for measuring contact angle of porous materials such as wood. (Brugnara, Volpe, & al., 2006; Bryne & Wälinder., 2010; Englund, Bryne, Ernstsson, Lausmaa, & Wälinder, 2009; Hakkou, Pétrissans, Zoulalian, & Gérardin, 2005). In the first receding curve or second cycle curves (both advancing and receding) this heterogeneity of the wettability does not exist anymore, because water has wetted the surface and contact angle between wood surface and water for these parts equals to zero. For octane curve, the first advancing curve is more linear and just an initial deviation can be observed, due to a fast initial wicking occurring at a depth of 2-4 mm (Wälinder & Johansson, 2001). As shown in Fig. 1., firstly, the final liquid uptake ( $F_f$  in Eq. 3.) for water is higher than octane, and secondly it increases during repeated cycles. This indicates that the sorption process for water in wood is more time-dependent. It is not surprising that water as a swelling liquid needs time to swell the cell wall of wood while octane only fills the voids and does not penetrate the cell wall, so its sorption takes place immediately after immersion of veneer and there is not so much liquid uptake afterwards.

The results of advancing contact angle of 10 different pine veneers are in range of 69-78° with average of 73±3° which is higher than values of other studies of pine veneers (Pétrissans, Gérardin, El Bakali, & Serraj, 2003; Winfield, Harris, & Hutchinson, 2001). Wang et al. (2007) got same range of advancing contact angle value for oven dried yellow pine.

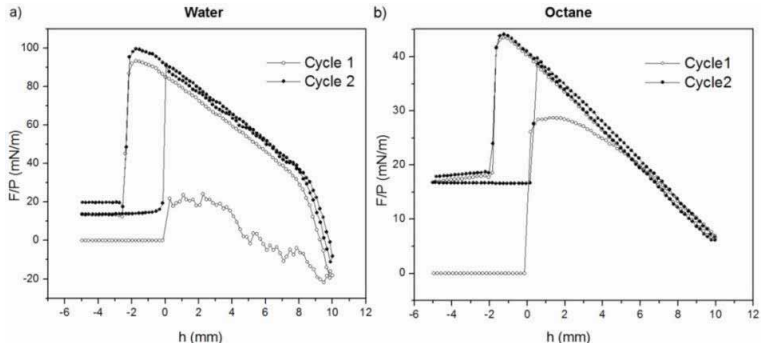


Fig. 1. Wettability measurements of pine wood veneer using Wilhelmy method given as force divided by perimeter as a function of immersion depth for (a) water and (b) octane on the same sample.

### Imbibition at constant depth (immersion to 5 mm and partial withdrawal to 2 mm)

In this method the veneer is immersed to a specific depth (5 mm) and then immediately withdrawn to shallower depth (2 mm). The contact angle between water and wood surface is zero during this measurement, Therefore no change in contact angle is observed in imbibition curves, see Fig. 2.. When a veneer is immersed in a liquid for a long time (e.g. 60 min.), the liquid swells the wood cell wall causing an increase in the veneer dimension. Since in this method, before starting the measuring force the sample is already immersed and has taken up some liquid, an initial force ( $F_i$ ) is observed in imbibition curves which could demonstrate as a preliminary absorption (Fig. 2.). This preliminary absorption is much higher for water than octane, and water could swell more by time. We can obtain the rate of wettability and penetration of liquids to wood sample by using this approach. As mentioned before, initial absorption data could be measured in this method as well.

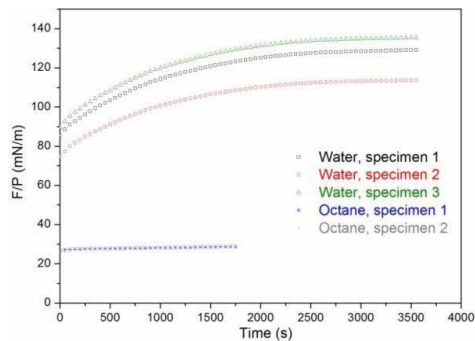


Fig. 2. Results of imbibition at constant depth for 3 pine specimens in water and 2 pine specimens in octane. The samples have been immersed to 5 mm depth and then withdrawn immediately to 2 mm depth. Results have been normalized by dividing by sample perimeter



### **Immersion to constant depth (without withdrawal)**

In this approach also the veneer is immersed to a specific depth (2 mm) same as previous method but this time, it is not withdrawn to shallower depth and absorption is studied by measuring liquid uptake in time just after immersion to desired depth. Three kinetic regimes are observed for water: (i) the fastest one with decreasing contact angle from about  $70^\circ$  to  $0^\circ$  which occurs at first 30 s of measurement time, (ii) the moderate regime which indicates filling the voids. This regime observes in the period of 30-180 s of measurement and (iii) the slowest one is tentatively associated with liquid up-take by swelling and diffusion (Fig. 3.). For octane two kinetic regimes are also observed. The first fast regime is due to initial wicking or same as water is because of filling the voids of wood. As octane has contact angle of zero with wood surface, we do not see any regime like water because of decreasing in contact angle in octane curves. After 30 s of measurement, the slower changing in weight for octane case can also be observed. The long-time changing with octane is significantly slower than for water.

Wang et al. (2007) used immersion method for 100 s with an immersion depth of 4 mm as a liquid absorption technique on wood samples. Our results show that 100 s is too short for studying liquid penetration due to existing some time-dependence regime in this type of absorption study. This method can be used to investigate the imbibition rate of wood samples by both swellable and non-swellable liquids. But for liquids with contact angle of higher than zero, the first kinetic regime which is due to decreasing the contact angle, might conflict the other regimes and make errors.

### **Full immersion**

Another way to prevent contact angle changing during measurement is full immersion technique. The veneers are horizontally immersed in probe liquid very quickly and liquid absorption study is performed in desired time. There is no longitudinal liquid transportation because both end grain sides of veneers are coated by a PU lacquer. A time-dependent force,  $F_w(t)$ , and final liquid uptake ( $F_f$ ) can be obtained from this method. Fig. 4. shows that, water penetrates more than octane into the wood. From curves, two features can be seen: firstly a slow uptake of water which occurs after a fast initial sorption and secondly sudden jumps in water curve that we interpret as sudden bursts of air leaving the sample.

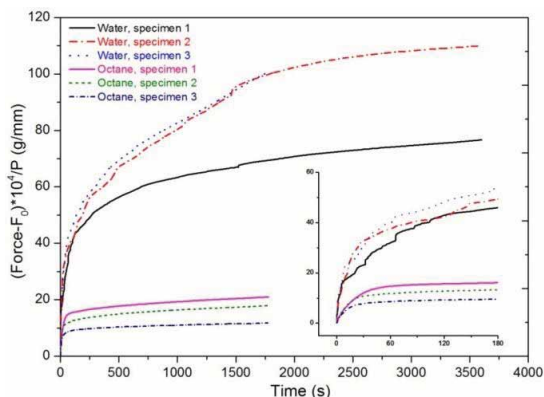


Fig. 3. Results of immersion to constant depth for 3 specimens in water and 2 specimens in octane. The samples have been immersed to 2mm depth and liquid uptake has been recorded. Results has been normalized by subtracting with primary force and dividing by sample perimeter. Smaller curves show the initial results of the same samples

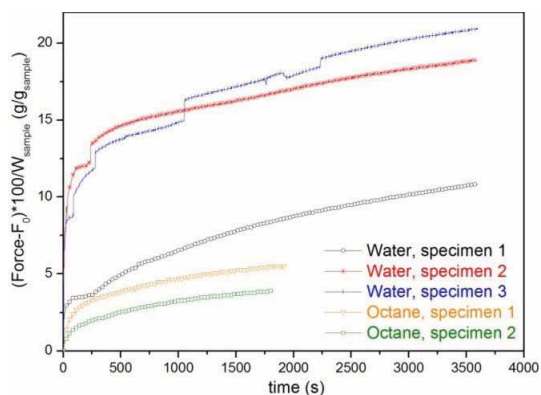


Fig. 4. Results of full immersion measurement for 3 specimens in water and 2 specimens in Octane. Results has been normalized by subtracting with primary force and dividing by sample weight

## CONCLUSIONS

This study shows that contact angle results from Wilhelmy method are reproducible. Water as a swellable liquid showed larger penetration and absorption than a non-swellable liquid like octane. Not only it presents initial fast wicking like octane, but also a time-dependence sorption process, swelling, is occurred in water absorption study. Comparing different absorption methods with Wilhelmy confirmed that imbibition in constant depth has a minimum number of kinetic regimes in data, and it makes understanding of wetting curves much easier.

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# APPLICATION OF HIGH-FREQUENCY DENSITOMETRY TO DETECT THE CHANGE OF EARLY- AND LATEWOOD DENSITY OF OAK (*QUERCUS ROBUR* L.) DUE TO THERMAL TREATMENT

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## ABSTRACT

The heat treatment of wood is an environmentally friendly method for wood protection. The progressive thermal degradation of the wood components at process-temperatures above 180°C leads to the reduction of hydroxyl groups. For this reason hygroscopicity, dimensional stability and durability are improved. In addition, heat treatment results in varying weight loss, depending on the treatment parameters, such as maximum treatment temperature, heating rate, holding time at the maximum temperature and the medium in the atmosphere, such as water vapour, oil or nitrogen. The increasing mass loss leads to progressive decrease in density. Here, the early and late wood growth zone of a tree ring can be altered to varying degrees. This ultimately affects the properties of the thermally modified wood. Therefore the aim of the present investigation was to detect the change of density of oak (*Quercus robur* L.), due to the influence of a thermal modification. The reduction of the density was recorded by High-Frequency Densitometry and compared to measurements based on the Gravimetric Method. To achieve a comparison of the initial density before and the density in result of thermal treatment, the examination was carried out on the same sample. The evaluation of the methods allows to conclude, that the High-Frequency Densitometry Method shows less deviations in their results than the Gravimetric Method. In addition this method provides reasonably good results in respect to the intended localisation of the reduction of density within an annual- ring. As to be expected, in contrast to the Gravimetric Method the High-Frequency Densitometry Method proved to be applicable to localise the change of the tree-ring density. It can be summarized that the measurement of the

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local density change is possible and the density of earlywood was significantly more reduced compared to that of the latewood. In parallel, changes occur in the microstructure in the form of cell wall delamination.

Key words: High-Frequency Densitometry, wood density, Thermal Treatment.

## INTRODUCTION

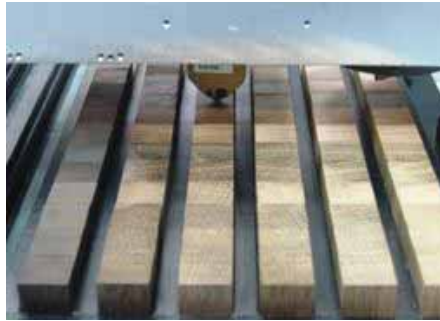
The thermal treatment of wood is an environmentally friendly method for wood protection. This treatment results in varying weight loss, depending on the treatment temperature and exposure time (Ates *et al.* 2009). Depending on the treatment parameters, such as maximum treatment temperature, heating rate, holding time at the maximum temperature and the medium in the atmosphere, such as water vapour, oil or nitrogen, cracks can appear in the material and the cell structure is partially degraded as well (Wagenführ *et al.* 2005; Kocaefe *et al.* 2007). These factors effect on the changes in wood density. Beside strength, hardness, calorific value and stability a range of other physical, mechanical and technological properties of wood are determined by the density of the material (Niemz 1993). The physical and mechanical properties of latewood are 1.5-2 times higher than earlywood. The more pronounced the content of latewood in an annual ring, the higher are the mechanical properties of wood.

Both, the High-Frequency Densitometry (HFD) and Gravimetric Method (GV), are suitable to detect the changes of density. The purpose of this approach was to determine whether the HFD –Method provides useful advantages in locating the changes of density. The investigation will therefore answer the question, whether the effect of thermal modification occurs to varying degrees, evenly throughout the growth area of a tree ring or just in certain areas with different types of tissue.

## MATERIAL AND METHODS

The investigations were carried out with wood samples of the species oak (*Quercus robur* L.) The samples were planed and sawn into specimens in direction parallel to the grain. Than they were cut into specimens with a dimension of 20x20x20 mm (tangential x radial x longitudinal). The overall amount of 48 specimens was assembled by choosing 12 specimens, arranged behind one other, from each sample. In an upstream step of preparation all these “twin samples” were arranged in a special pattern and calibrated to achieve a high quality cross-section surface, for the measurement of density by HFD- Method. At the beginning the wood samples were conditioned to 7-8% equilibrium moisture content at 20±2°C and 65% relative humidity. The Gravimetric Method refers to the determination of density according to the DIN 52182 (1976). In the next step the HFD- Method was applicated to measure the density of the identical specimen as mentioned above (Fig. 1). The method is based on the propagation of continuous electromagnetic waves in a high-frequency (HF) transmitter-receiver link of an extremely small electrode system in direct contact with the wood

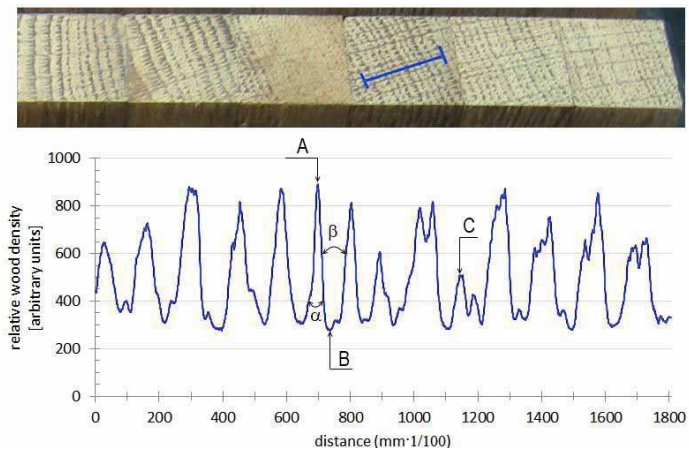
surface that is to be investigated (Schinker *et al.* 2003). In the next step the specimen were treated in a temperature controlled heating unit at three different temperatures (180, 200 and 220°C) with 6 hours duration. The process conditions were applied and run under atmospheric pressure and in the presence of nitrogen. Nitrogen was pumped into the chamber at 124°C to prevent the decrease of hemi-cellulose and the linked loss of mass of the specimen. After treatment, the temperature was reduced within 24 hours to 20°C before the specimens were conditioned (20°C, 65% relative humidity). The same procedure was run for all experiments, before the dimensions and weights were measured.



**Fig. 1** Process of density measurement using a highly sensitive sensor for wood surface scanning (RINNTECH® technology)

## RESULTS AND DISCUSSION

As a result of HF-Densitometry early and latewood density values were obtained, as shown in Fig.2. The curve describes the local density on the interval length of 18mm (blue line). All measurements were carried out in radial direction, by arranging the measurements perpendicular to the annual ring.



**Fig. 2** Wood density profiles of oak (blue line) measured by HF-Densitometry

The density of latewood is characterised by the points of maximum on the density curve (point A in Fig. 2). In contrast to this the earlywood density occurred as the points of minimum, as shown in point B in Fig. 2. It should be emphasized also the obvious difference in the ratio of measure between  $\alpha$  and  $\beta$  angles; as one can see  $\beta$  angle exceeds the angle  $\alpha$ , as well as earlywood growth is greater than the increase of latewood, which is quite natural. Furthermore it should be noted that the presence of abnormal values on the density curve are difficult to explain, just by statistical scattering or based on the scientific approach. For example the value of density at point C (Fig. 2) is very low for oak latewood. According to the authors, this can be explained by insufficient preparation of wood surface, which led to the measurements of air. In such a case contact force between the sensor and the surface is inadequate, which leads to errors in measurement. The range of specimen would need to be widened to improve this statement. In Fig. 3 the curve of density before and after thermal modification is plotted according to the treatment intensity of a) 180, b) 200 and c) 220°C.

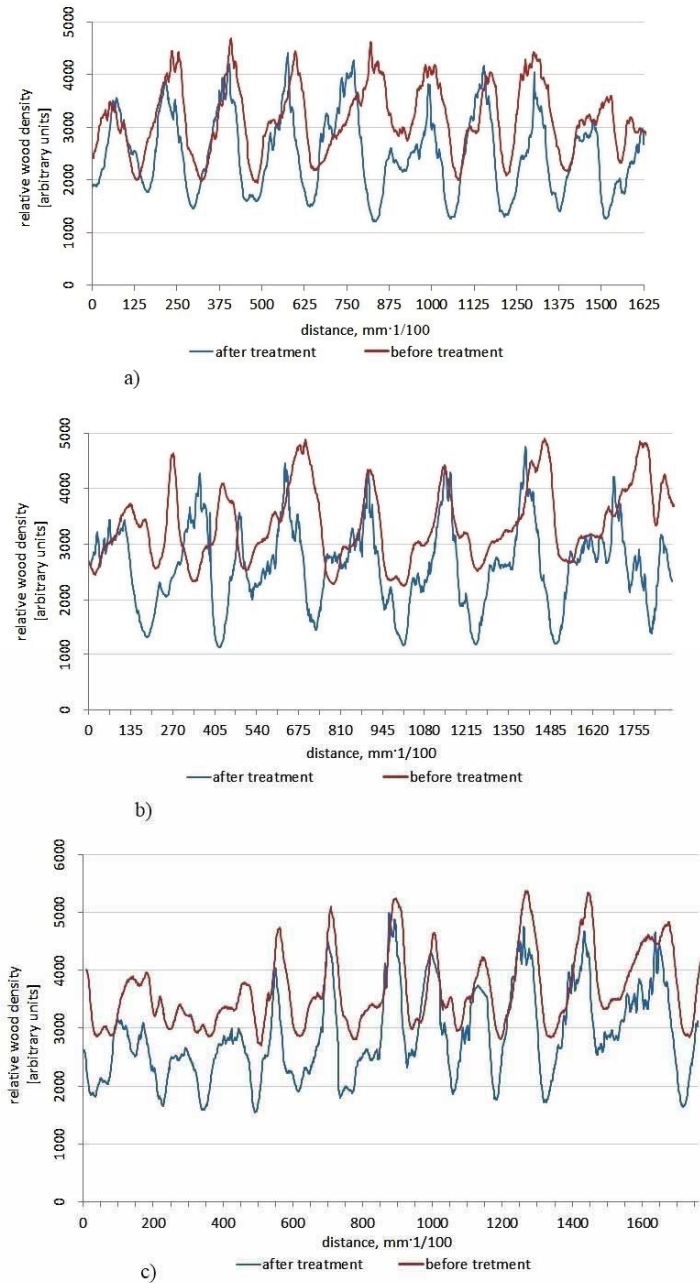
Relative to the latewood section of the annual ring, the earlywood section with its larger volume and its differing microstructure shows higher sensitivity in respect to the height of the temperature. This peculiarity can be explained by the anatomical and chemical structure of wood and its subsequent reaction due to the heat treatment (Thomas *et al.* 2007). The degradation of the tracheid walls during heat treatment (Wälinder *et al.* 2009) and the degradation of the hemicelluloses (Pfriem *et al.* 2009), which are one of the major components of the cell wall, is reflected in the reduction of the overall density.

Comparing the change of density it can be stated that the density of earlywood decreased by 12.3% while the one of latewood only by 4.3% at a temperature of a) 180°C. However, as a result of a treatment at c) 220°C a maximum reduction of 20.6% for earlywood and 12.7% for latewood has been achieved. In addition to the statement that increasing temperature of thermal modification is provoking wood density decrease these results indicate the proportion of reduction in earlywood as 2-3 times higher than in latewood.

In a comparison of the mean values for the decrease in density it is apparent that the degree of reduction, for both methods is approximately the same size. Values, which are measured based on HFD-Method show reduction from 8,5% at 180°C, 11% at 200°C and 16% at 220°C, while values measured with the Gravimetric Method are 6,5% at 180°C, 10% at 200°C, and 14% % at 220°C.

But comparative analyses show that the absolute measured values are smaller for the density by the HF-Densitometry, as the results of Gravimetric Method. The difference is in the early- and latewood equal by 18%. According to the authors, this is due to shortcomings of both methods, such as using inaccurate measurements for the Gravimetric Method and insufficient quality of surface preparation for HF-Densitometry.





**Fig. 3** Wood density profiles of oak measured by HF-Densitometry before modification and after treatment at a) 180°C, b) 200°C, c) 220°C

## CONCLUSIONS

Thermal modification of oak (*Quercus robur* L.) leads to a decrease in density, as a result of an increasing treatment temperature. In contrast to the Gravimetric Method, the HFD- Method allows to differentiate the noticeable changes, concerning earlywood density with e.g. up to 21% and latewood density reduced by 12%. The evaluation of the methods allows to conclude, that the High-Frequency Densitometry Method shows less deviations in their results than the Gravimetric Method.

This results lead to the conclusion, that in further studies locally resolved measurements of topochemical changes should be added. The localized changes in density could be adjusted for example by NIR spectroscopy, which can provide spatially resolved measurements with information about changes in the molecules-structure. Further experiments on wood species, with more clearly defined early- and latewood, are part of further research.

## ACKNOWLEDGEMENTS

This research has been conducted in cooperation between the Eberswalde University for Sustainable Development and Ukrainian National Forestry University with the support of the German side. The authors would like to thank Nicole Linke and Prof. Dr. Martin Guericke for providing the HFD equipment, Christian Müller for his assistance in conducting experiments and preparing the experimental samples as well Nadine Herold and Fabian Wulf for active participation in the discussion of the results.

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# INVERSE GAS CHROMATOGRAPHY CHARACTERIZATION OF WOOD COMPOSITE COMPONENTS

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## ABSTRACT

Inverse gas chromatography (IGC) can be used to study the surface characteristics of individual wood, plastic and additive components prior to processing into wood-thermoplastic composites (WPC). Such information may be helpful to tailor and improve the molecular bonding and stress transfer capacity between the reinforcement and matrix in order to enhance the resulting durability and mechanical performance of the completed composite. The main feature of IGC is surface energy characterization of materials in particle, fibrous or powder form, e.g. wood processing byproducts. This paper covers some background information about the IGC technique as well as some initial trials to determine surface energy heterogeneity of some ground wood and micro crystalline cellulose components of different size distributions.

Key words: Inverse gas chromatography, wood plastic composites, surface energy characterization.

## INTRODUCTION

Inverse gas chromatography (IGC) is particularly useful to determine surface free energy and acid-base characteristics of materials in particle or fibrous form [1, 3–9]. Recently, because of the increasing use of wood plastic composites (WPC), a growing interest can be noticed especially regarding application of the IGC technique to study the surface characteristics of wood and also its interaction with thermoplastics [1, 3, 4, 6, 7, 9]. From these studies it is clear that the application of the IGC technique to study the surface energy characteristics of the matrix and reinforcing materials used for WPC

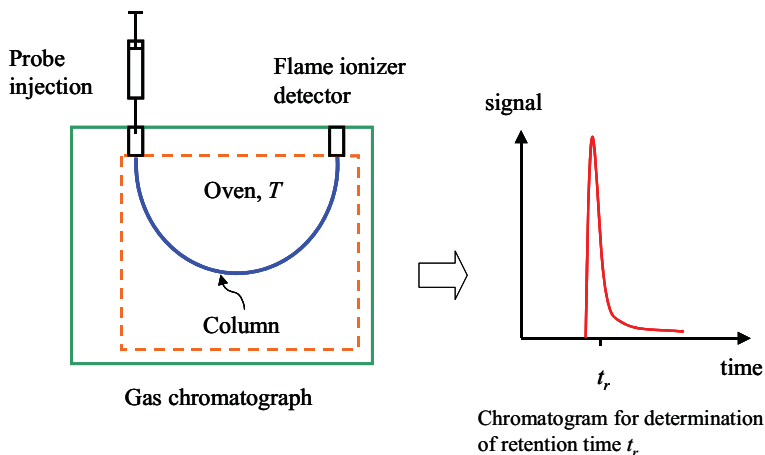
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adds valuable insight about their internal bonding properties, i.e. the compatibility between the wood and thermoplastic components.

The principal of IGC is like regular gas chromatography where gases are injected into an inert gas stream passing through a packed column held in an environmentally controlled oven. As the gases reach the end of the column, they are detected using a flame ionization detector. In normal gas chromatography, the columns are chosen for their ability to separate gases adequately, whereas in IGC, known gas probes are chosen for their ability to interact with an unknown packing material. Figure 1 shows a sketch of the IGC principle.



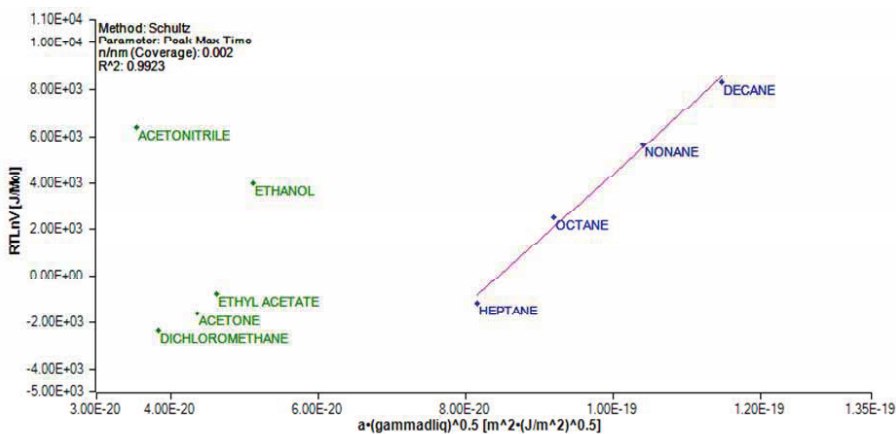
**Fig. 1.** Schematic illustration of the principle for inverse gas chromatography (IGC) experiments

The objective of this work is to present some background information about the IGC technique as well as to present an initial trial to determine surface energy heterogeneity of some wood components with different size distributions.

## MATERIALS AND METHODS

Two different in-house experimental ground softwood samples were investigated, one with a course and one with a fine average size, see description in Table 1. In addition, for comparison reason, a sample of commercially available micro crystalline cellulose (MCC), Avicel PH-101, was also included in the study. The samples were packed in 300 mm long glass columns with 6 mm outer diameter and 4 mm inner diameter. The column packing procedure involved a sample holder and packing device supplied by Surface Measurement Systems (SMS, Alperton, London, UK). Glass wool was placed in both column ends to hold the sample material in place.

IGC was performed on a state-of-the-art IGC Surface Energy Analyzer (SEA) developed by Surface Measurement Systems, Alperton, London, UK. The IGC experiments were performed at both infinite (low surface coverage) and finite (higher surface coverage) dilution using a series of non-polar probe gases (n-alkanes). The finite dilution experiment enables determination of both the BET specific surface area as well as the surface energy variation on the sample, i.e. the heterogeneity of its surface energy [8]. See Figure 1 for the principle experimental set-up, and Figure 2 for an example analysis plot of adsorption (or retention) of a series of *n*-alkanes (ref. line) and a series of acid-base probes on the wood component sample. Further descriptions of the fundamentals of IGC technique can be found in references [2, 8].



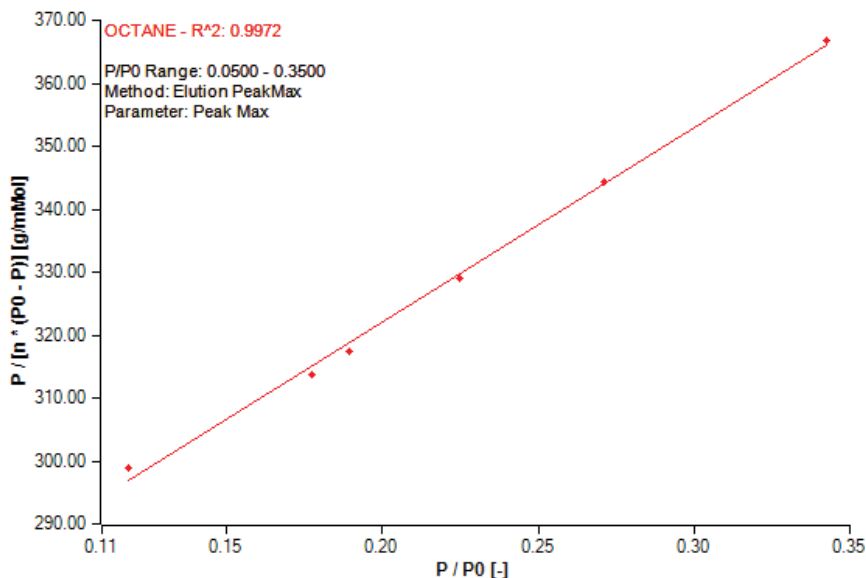
**Fig. 2.** Example plot of the retention volume ( $RT \ln V_N$ ) versus the molecular descriptor, i.e. the polarisability index (horizontal axis) for adsorption of a series of *n*-alkanes (ref. line) and a series of acid-base probes on the coarse wood component sample

Purified helium was used as the inert carrier gas and the flow rate was 10 mL/min. The instrument injects a finite amount of vapour which produces the resulting chromatograph from which the retention times can be calculated. A SMS software was used to determine the retention time of the various probe chromatograms relative to that of methane, which was used as a reference gas probe. The experiments were performed with an oven temperature held at 30 °C.

In the IGC experiments it is essential to initially remove as much water and volatiles as possible from the particles, and therefore, the samples were conditioned for 4 hours in the gas chromatograph at 80 °C. A series of non-polar *n*-alkanes (heptane, octane, nonane and decane) and polar acid-base (ethanol, dichloromethane, acetone, acetonitrile, ethyl acetate) anhydrous probes were used in the IGC analysis. All liquids used were of high-performance liquid chromatography (HPLC) grade (suitable for chromatography analyses) and purchased from Sigma-Aldrich.

## RESULTS

Figure 3 shows a plot for the determination of BET surface area of the coarse wood component sample using octane as probe gas. Table 1 presents obtained BET specific surface area of the three samples. It is noteworthy that the fine wood component sample proved to have the highest specific surface area (1.6 m<sup>2</sup>/g), followed by the Avicel and the coarse wood component samples (1.2 and 0.7 m<sup>2</sup>/g, respectively).



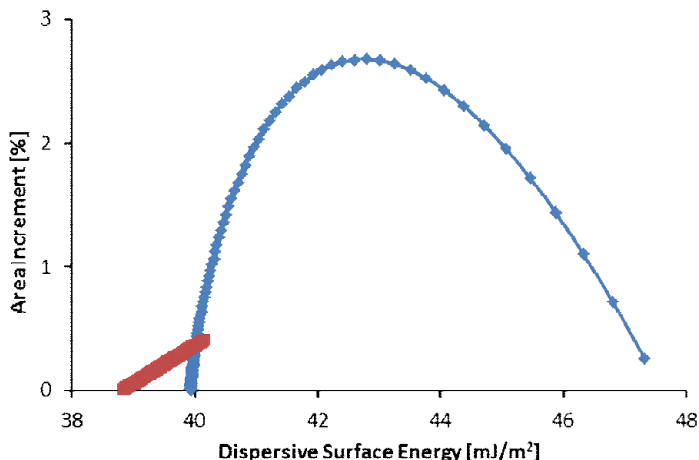
**Fig. 3.** Plot for the determination of BET surface area based on the adsorption octane at different surface coverage (or partial pressures  $P/P_0$ ) on the coarse wood component sample.

**Table 1.** Description of the ground wood component samples and MCC including the determined specific surface area.

Material	Description	BET Surface Area [m <sup>2</sup> /g]
MCC	Avicel PH-101, average size 50 μm	1.2
Fine wood component	Softwood, average size 30 μm	1.6
Coarse wood component	Softwood, average size 750 μm	0.7

Figure 4 shows a plot of the dispersive surface energy distribution of the two wood components. As can be seen, the finer sample has a distinctly different distribution, i.e. a straight line, compared with the parabolic shaped curve for the coarse sample. A possible explanation of the straight line distribution of the fine component could be that these sample is more homogeneous compared with the coarse component. It was, however, not possible to obtain any reliable corresponding data for the Avicel sample,

probably due to poor method settings for the surface coverage alteration. At this stage, no methods for acid-base analysis were developed.



**Fig. 4.** Surface energy distribution of the two different wood components (red/left line: the fine wood component; blue/right curve: the coarse wood component).

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# WOOD – ENERGY, EMISSIONS, EXPERIENCE (WEEE)

Tellnes, L. G. F.<sup>1</sup>, Nore, K.<sup>2</sup> & Nyrud, A. Q.<sup>3</sup>

## ABSTRACT

A substantial share of global energy consumption is used to accommodate desired indoor climate. Increased focus on energy efficient buildings has resulted in rising concerns about possible adverse effects on human health. The objective of the research project WEEE (Wood – Energy, Emission, Experience) is to document the properties for solid wood as a healthy and energy efficient building material. The purpose of this paper is to present relevant issues investigated in the project.

The introduction of construction classification schemes is likely to increase the importance of building material emission labelling. These labels often depend on the indicator for emissions of total volatile organic compounds (TVOC), but the use of this indicator has been disputed. Natural emissions levels from solid wood show considerable variations between species and with different samples of the same species. Terpenes are the largest part of these emissions, but there is little knowledge about the contribution of individual compounds on the indoor air quality (IAQ). However, there are studies indicating that terpene emissions mixed with ozone will react into compounds that cause sensory irritation. Further work in the project will therefore be to measure continuous emissions from Norwegian softwood (Norway spruce, *Picea abies* L. Karst and Scots pine, *Pinus sylvestris*) in a full scale indoor climate laboratory with exposure testing on volunteers.

Key words: Energy, emissions, experience, health

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## INTRODUCTION

In Norway, buildings account for approximately 37 % of the energy consumption (Report No. 28 to the Storting, 2011-2012) and a substantial share of this is used to accommodate desired indoor climate. However, the rising focus on energy efficient buildings is accompanied by high concern to indoor climate and health issues. The objective of the WEEE project is thus to: ‘Provide scientific evidence of wood’s suitability for construction of energy efficient and healthy buildings’.

The scope and parts of energy, emissions and experience are illustrated in Fig 1. With reference to WP1 Energy, this work will focus on discovering the potential energy savings from heat buffering of solid wood and the advantage of moisture buffering. The potential effects of latent heat exchange are especially in focus in WP1. The emissions from solid wood have a large natural variation and can take longer time to decline than several other building products. Energy and emissions are both important parts with reference to the experience of the indoor environment made of wood.

## WEEE - Scope

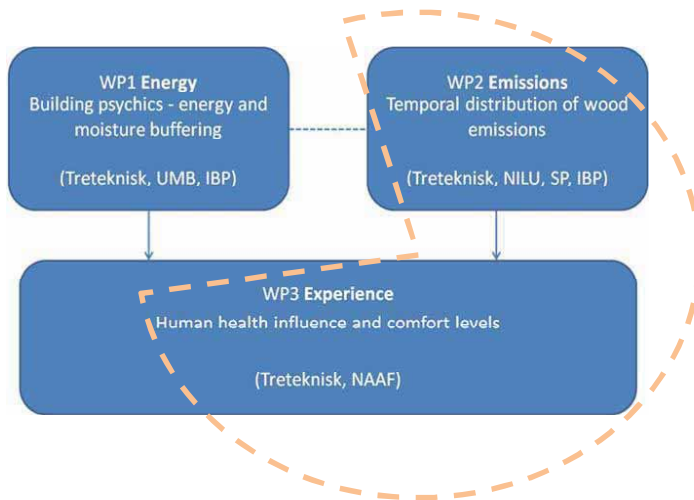


Fig. 1: Scope of the WEEE-project and the scope of this state-of-the-art paper.

The scope of this paper is narrowed down to the link between emissions from solid wood and health effects. This is illustrated with the plotted line in Fig. 1. This issue covers the release of volatile organic compounds (VOCs) from wood and effects on human health and comfort with sensory irritations and odour. Wood has been considered a natural interior building product for many years and is not known to cause any serious health concerns and have therefore be regarded as a safe material.

However, there have been some experiences with sensory irritations that has been assumed to be caused by the emission of terpenes from pine.

## PURPOSE

This review has investigated studies on emissions from untreated solid wood products of softwood and studies on potential health issues from emissions from building products. Also, standards and metrics that are relevant for this issues have been review. This paper will cover both the scientific basis for the relationship between emissions and health effects and how this is practiced in metrics used to document and evaluated building materials is covered.

## BUILDING CLASSIFICATION AND EMISSION LABELS

In Norway, a national adaption of the international BREEAM classification have been developed into BREEAM-NOR. The interest of the construction industry and the demand from large actors within property management have been important along with a early inclusion of stakeholders has ensured a wide anchoring. The international version limited the criteria to formaldehyde emission from wood panels, wood floors and glued laminated timber. BREEAM-NOR also include criteria for materials classification of low polluting materials as in NS-EN 15251:2007 Appendix C, which is based on the same measuring criteria as in the finish Emission Classification of Building Materials (M1) (NGBC, 2012). This criteria is also to be used for environmental product declarations (EPD) of solid wood products in Norway. However, it is voluntarily to include this information. In the relevant EPD on Norwegian interior wood panels, it was not measured (Grini, 2010).

The requirements of M1 is that the emissions of TVOC should be  $<200 \mu\text{g}/\text{m}^2\text{h}$ . But in the related Finnish Classification of Indoor Environment, softwood can be used without limitation and still receive the best ranking S1, despite that this demands only use of M1 ranked materials (Säteri, 2008). Mølhave (2009:336) introduced the TVOC as a result of 'not knowing what to do'. It was emphasised that it was only intended as a screening method for sensory irritation. Some of the issues discussed was the different procedures for TVOC measurements and the lack of established dose-response relationships.

The increasing use of building classification schemes like BREEAM-NOR are likely to increase the importance of emission labelling like M1. The challenge could be that the TVOC indicator that is originally only intended as a screening indicator, will be determining the choice of materials for buildings that are to be classified with BREEAM-NOR.

## EMISSIONS FROM SOLID WOOD

Englund (1999) measured emissions of volatile organic compounds from 41 material samples from pine (*Pinus sylvestris*), spruce (*Picea abies*), beech (*Fagus sylvatica*), oak (*Quercus robur*) and birch (*Betula pendula*), all of Swedish origin. From all the wood species, large individual differences in the emissions from parallel specimens were found. This variation is influenced by climate at the growth place, nutrient supply, genetic factors, time of harvesting, drying and handling of the timber. Average values for emission rates of each wood species are therefore not desirable or meaningful to establish. The emissions from pine after 28 days vary from 500 to 4800  $\mu\text{g}/\text{m}^2\text{h}$  and the dominating compounds were  $\alpha$ -Pinene and 3-carene.

In Emissions from Wood-Based... (1998), the emissions from pine and spruce were evaluated against the M1. The results showed large variations, but the general indication is that spruce is under the threshold for TVOC in M1 and pine can have up to ten times higher than the TVOC threshold.

## INDOOR AIR QUALITY AND HEALTH

In 1997, a Nordic workshop was held to develop consensus on the understanding of possible health implications by emissions from wood products. One of the main conclusions was that 'no indications have been shown that emissions from solid wood, used as intended, cause health effects such as allergy, carcinogenic, neurotoxic and reproductive toxic effects in the indoor air'. It was recommended that further work should look on houses and rooms with relatively high wood-lead. Additionally, it was stated that the reactions with oxygen and ozone with the emission from wood in air might be of major importance, but by implications not yet explained (Emissions from Wood-Based..., 1998).

Andersson et al. (1997) presented results from a literature review by 12 Nordic researchers on the issues of VOC emissions and health effects. The conclusion was that indoor air pollution including VOC is most likely to cause health effects, but that there is no support for the use of TVOC as a risk parameter. Hence, TVOC should not be used as a limit value for air concentrations and emissions from building materials.

ECA-IAQ (2007) is a state of the art for ozone-initiated chemistry and IAQ. It stated that so far, only formaldehyde have been identified as a source for sensory irritants. Recent work found it likely that reactions of terpenes and ozone are also a cause for sensory irritations through the formations of radicals such as OH, HO<sub>2</sub> and RO<sub>2</sub>, and stable products as aldehydes, peroxides and condensed phase compounds. Some of these are sensory eye and airway irritants, but many of these cannot be measured by traditional analytical techniques. Terpenes are widespread in nature and particularly conifers. Ozone is a common compound in outdoor air, but there are few sources indoors. Ozone occurs in the indoor air mainly because of air exchange, but deposition on materials are important for enhanced levels.

Nyrud, Bringslimark & Englund (2012) have assessed the potential effects of use of solid wood panels in patient room with its VOC emissions. The conclusion was that the wood panels had little contribution to the VOC levels in the rooms. Gminski et al. (2011) have tested for sensory irritations and pulmonary effects from short-term exposure to pinewood emissions on healthy volunteers. Even with very high concentrations, no effects were found except odour which in general was rated as 'pleasant' rather than 'unpleasant'.

## CONCLUSIONS

- The use of emissions labels in building classification is likely to increase the demand for emissions labelling.
- TVOC is the common threshold for emission evaluation of building materials. The reason is correlation with sensory irritations, but it was only intended as a screening indicator and its use have been heavily debated. However, in the lack of alternatives it is still used.
- Sensory irritation caused by VOC emission seems to be caused by products of reaction between terpenes and ozone indoors.
- Area-specific emissions are not ideal for measuring potential health effect from VOCs in indoor air due to secondary reactions in the indoor air.
- Further investigations on health effects from emissions from solid wood should focus on testing sensory irritation in combination with continuous measurements of compounds in indoor air.

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# INVESTIGATING LATENT HEAT EXCHANGE OF UNTREATED WOOD PANELS

Brueckner, C.<sup>1</sup>, Nore, K.<sup>2</sup> & Nyrud, A. Q.<sup>3</sup>

## ABSTRACT

Wood may be a conditioning component in the indoor environment because the pore structure of wood interacts with the ambient air. These interactions between indoor climate and building interiors may improve energy efficiency as well as perceived comfort for occupants. The phase transition of the vapor in air to bound water in the wood cell wall emits energy, and an exothermic reaction occurs. This experiment is part of the research project WEEE, Wood - Energy, Emissions, Experience coordinated by the Norwegian Institute of Wood Technology. The experiment is carried out in a controlled climate through 24 hour periods with cyclic air humidity. Surface temperature of spruce and pine panels are measured by thermography and temperature sensors and the moisture uptake is logged by weighting cells. This study provides valuable insight on the influences of latent heat exchange in buildings. When untreated wooden cladding is used, the latent heat should be included in the energy balance.

Key words: Latent heat, thermography, indoor climate, wood, spruce, pine, untreated panel

## INTRODUCTION

The main objective of the project WEEE, Wood- Energy, Emissions, Experience, is to investigate woods' suitability for the construction of energy efficient buildings which provide a healthy environment for their occupants. The project is divided in three elements which examine energy, measure emissions and occupants' experience. The energy part intends to measure potential energy savings in a wood environment.

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Latent heat exchange for untreated Norwegian Spruce (*picea abies*) and Scots pine (*pinus sylvestris*) are investigated by cyclic changes between high (90%) and low (20%) relative humidity (RH) with 12 hour steps and analyzed by thermography. The influence of the annual rings and cellular direction to thermal energy was also investigated.

Korsnes, S. K. (2012) summarized correlations between air, moisture and heat exchange on indoor surfaces and explained the complex correlation between moisture, heat and air flow. For porous materials with some moisture content (MC), the temperature on surfaces is defined by evaporation and condensation. Evaporation and condensation is strongly influenced by air velocity, air temperature and moisture difference between air and surface. Fig. 1 shows the physical reactions and dependencies of porous surfaces according to material properties and ambient air climate during absorption.

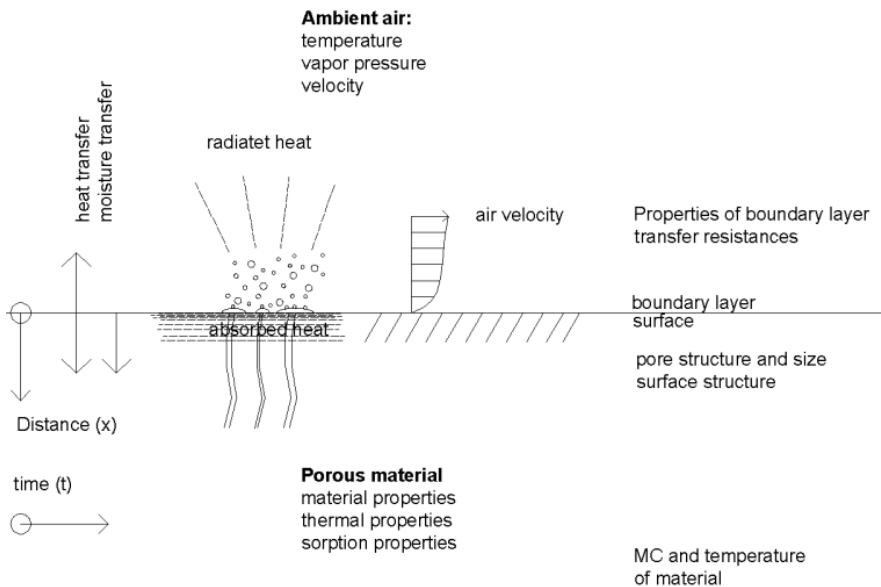


Fig1: heat and moisture transfer between wood, boundary layer and ambient air

The hypothesis is; what is woods potential to save energy due to its latent heat of sorption?

Using wood as surface material has both aesthetic and technical advantages. Wood has, due to its capillary and porous structure, an advantageous physical structure for dampening water and temperature cycles. Wood counteracts large changes in order to keep the vital moisture supply. Wood absorbs and condensates water. The phase change from damp to bound water, radiates sensible heat. This heat may increase the

surface temperature several degrees. All interiors of untreated wood will react to the natural diurnal RH and temperature fluctuations and can be incorporated in energy effective design. In low-energy houses where wooden surfaces are dominant it may help to fulfill strict energy regulations and increase comfort.

The porous structure of wood is an effective support for a long time latent heat reaction. Due to the water transport system of trees, water on the surface is soaked into the capillaries and bounded physically to the cell walls. The condensation on the pore surfaces proceeds until the tracheids vapor pressure equals the vapor pressure of the ambient air. Sorption speed differ between spruce and pine due to the high content of extractives in pine. Sapwood and heartwood are also likely to differ, because the fibers of heartwood are blocked from water transport with extractives. Different sorption behavior of early- and latewood occurs because of their different physical tasks in the trunk. Early wood tracheas' task is the water supply during the growth period after winter, whereas latewood tracheids have smaller vessels and absorb less water and at a lower rate and is mainly for stabilizing the tree. The awareness of these relations are useful to find appropriate wood species for indoor climate support.

In this experiment, thermography is tested as a method for measuring temperature differences on the samples. Thermography is used to detect the emitted magnetic waves from objects in the infrared spectrum and displays it as thermal images. Emissivity is defined as the ability of a body to emit thermal radiation compared to a black body and distinguish materials in a range of 0,00 to 1 [-]. Planed softwood reflects radiation with an emissivity of 0.86 while aluminum is an appropriate ambient material with a low emissivity of 0.02 at 25°C. Thermography has high sensitivity for temperature differences in areas and enable comparison of several spots at a time. Temperature is also measured with thermocouples for control. Ambient air velocity interacts with moisture uptake and is measured on the surface. Weighting cells measure the varying weight of test samples. The reference samples are covered to exclude moisture uptake.

## MATERIAL AND METHODS

### Samples

The species of Norwegian spruce and Scots Pine with same number of annual rings and a dimension of 205 mm length and 105 mm width were measured in this experiment. All samples were mainly heartwood. Equal surface texture on both sample and reference was desired. Pairs of samples were cut out of tangential sawn panels and split in two halves to obtain mirrored surfaces for optimal comparison. The face sides were only planed once before the thickness of 5 mm was worked out from the backside. To avoid cupping and bending the panels were conditioned to the defined MC of 19 weight%. The different angles of the pith rays to surface as well as the early- and latewood were considered when analyzing temperature difference.

The sensors were placed as described in Table 1:

Table 1: Placing of thermocouples and defined areas by thermography.

Sensor	S1	S2	S3	S4	S5	S6
Sample description	Reference	Reference	Reference	Exposed	Exposed	Exposed
Local sample structure	Early wood radial	Early wood tangential	Latewood tangential	Early wood radial	Early wood tangential	Latewood tangential
Directly comparable to	S4	S5	S6	S1	S2	S3
Allocated area of thermography	Ar1	Ar2	Ar3	Ar4	Ar5	Ar6

The left-mounted samples were covered in transparent Low-Density Polyethylene (LDPE) foils which barely change the emissivity and has nearly no moisture uptake. The pairs were covered at the remaining five sides with adhesive tape and LDPE preventing unrecordable moisture reactions.



Fig. 2: Housing, samples (covered left, uncovered right) and thermocouples mounted on the ceiling. In center the air speed meter is placed. View from the thermographic camera's position.

### Instrumentation

Surface temperature of each sample and housing as well as the weight of both samples were continuously measured every fifth second. Room temperature and average RH was logged every minute by the climatic chamber sensors situated close to inlet fan. Average air velocity and temperature 5 mm below the surfaces were measured every 20 minutes by an air velocity meter (resolution: 0.1 °C, 0.01 m/s; accuracy:  $\pm 0.3^\circ\text{C}$ ,  $\pm 0.015$  m/s, according to Technetics 2008) placed concentric between sample and reference. Six wired thermocouples type K ( $\pm 1.1$  °C according to [www.omega.de](http://www.omega.de))

2012) were fixed by staples on each sample to the center point, latewood and early wood, so that the sensor area was mostly laid by wood respectively PE foil on the reference sample, and even to the surface in order to decrease air temperature influence. The temperature on the enveloped housing was measured to detect temperature fluctuation when RH was rising and vapor heated the air.

The samples were held by weighting cells (linearity 0.05 % at 20 °C (Maywood Instruments, 1995)) which provided the sample and reference mass change. The graphs of latent heat exchange peaks were closely examined when temperature and RH varies.

A thermographic camera with a resolution of 240×180 pixels and thermal sensitivity of <0.045°C (Flir, 2012) recorded the experiment at a distance of 0.5 m. The software allowed plotting average temperature of areas near the sensors. The rectangles had same dimensions and were laid along the annual rings.

### **Climate chamber**

The experiment took place in a 41 m<sup>3</sup> dark climate chamber with ceiling-mounted ventilation. Alternating RH was defined with constant temperature and velocity. The wall and ceiling surface were aluminum plates, which are beneficial for thermography measurements due to the low emissivity of aluminum. The sample housing was of MDF covered with aluminum foil. The boxed ventilation in the ceiling, a 0.5 m<sup>2</sup> spot was found for optimal placing with an average air velocity of 0.15 m/s ± 0.05 m/s (Air velocity meter, VELOCICALC Plus, 2011). The experiment was mounted in the ceiling to avoid thermal buoyancy. Our pretests showed horizontal differences in surface temperature when samples were mounted on walls. Except the thermography camera and the grounded Pc-Logger all electrical instruments were installed outside the room to reduce the signal noise to a minimum level.

### **Methods**

The climate cycles, changing RH from 90 to 20% and vice versa were set in order to obtain results for further experiments simulating indoor climate. The RH at start was 90% at 20°C for a 12 hours period so that surface temperatures were equal and equilibrium moisture content (EMC) was achieved. Then RH decreased steady to 20% in a period of 12 hours. After reaching the lowest point of the run, RH rose at maximum speed to 90% in duration of 6 and 8 minutes. The fast RH-rise simulated a rapid moisture excess in bathrooms like showering and the slow RH-fall was adapted to daily drying in buildings while high temperatures outdoors. The cycle repeated for 3 times.

## **RESULTS**

The thermocouples used as control measurement confirmed the thermographic results in every point, but had a lower accuracy and are not shown in this section. The

correlation with thermography is 0.90. The air velocity was kept constant as mentioned in materials and methods. The climate chamber had an unpreventable temperature fluctuation at RH change of maximal 2°C due to the vaporizer. However the samples were subjected to identical conditions. An effect in weight due to the hysteresis effect could not be detected.

The temperature was set constantly on 19 °C measured in front of ventilation. Due to thermal byonancy air velocity meter obtained the desired air temperature below the sample surfaces of  $20.0 \pm 0.5^\circ\text{C}$ . Vapor air density of 90% at 19°C is identical to 20 °C and 85%. The climate cycles were not similar in each run. However the vapor density difference was  $0,28 \text{ g/m}^3$  at maximum between the species. This is considered low.

Fig. 3 shows room temperature and RH in a period of one hour with RH load at 0:30. Room temperature increased from about 19.0°C to 20.7°C. RH started to increase from 21.6% the maximum at 93.5% and after 1h the 90% was reached. Room temperature rose exactly until the point where the climate software switched to the next step.

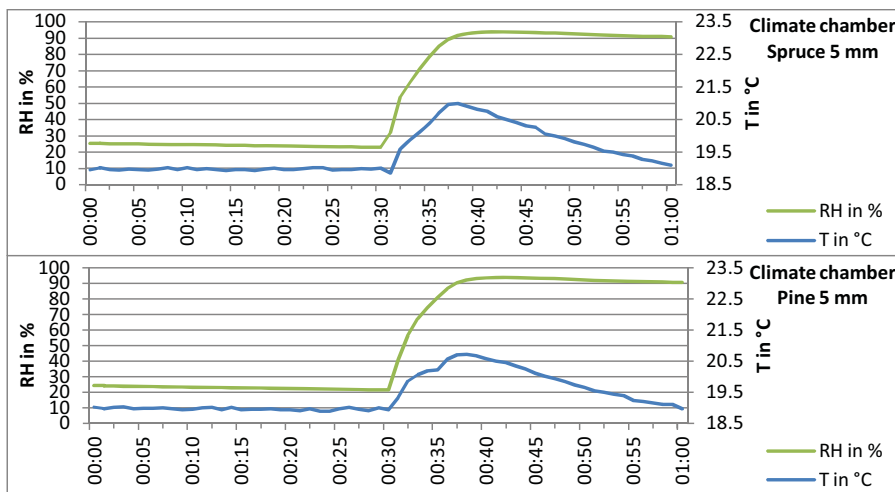


Fig. 3: Considered climate run of spruce and pine. RH (right axis) and room temperature (left axis)

In the plottet graphs in Fig. 4 a temperature rise in Ar4 of approximately 3°C of both samples throughout increasing RH is shown. The temperature gradients of both species seem to be identical, the water uptake of spruce went faster in the beginning.

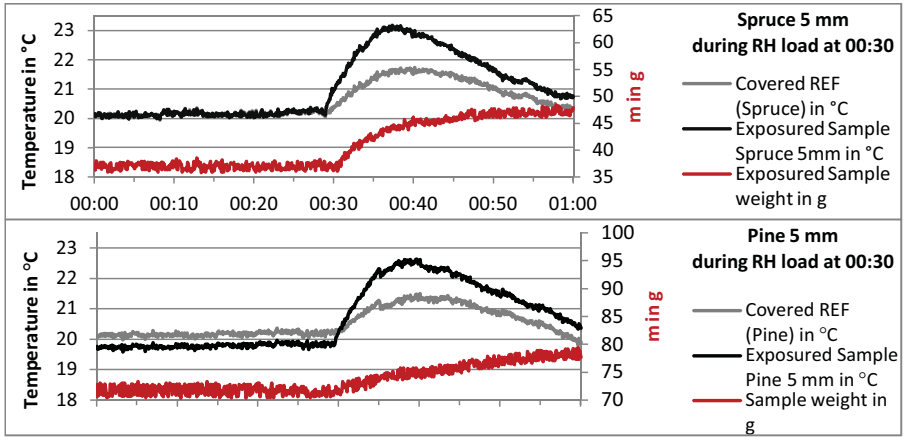


Fig. 4: Weight increase and thermal emission on spruce and pine during RH load.

The measured lower temperature on pine samples may be caused by emissivity deviation and were not closer examined. Low differences between the covered references Ar1 – Ar3 of only 0.04°C and 0.11°C on wood showed the influence of emissivity. The lowest temperature on species was nearly at any time in Ar6 with tangential cut latewood. This was confirmed by the thermocouples.

The temperature difference of spruce between areas of the sample and reference sample were from 0.4 °C to 0.6 °C at the begin of step 3 and rose faster than pine in a range of 1.5°C to 2.0°C after 7 minutes. Ar4 kept the temperature after 2 hours still 0.40°C higher than the reference sample. At this point weight and room temperature was constant. Temperature of reference samples increased by 1.0°C ±0.25°C due to room temperature rise and vapor condensation on the surface.

On Pine the highest peak was reached after 11 minutes in Ar4, Ar5 and Ar6 with differences to the comparable reference areas of 1.1°C to 1.2°C. After one hour while the RH decreased to desired value 90%, differences between Ar4 and Ar1 was still 0.5°C whereas Ar6 to Ar3 was 0.27°C. After 2 hours without RH rise, Ar4 was still 0.25°C higher than the remaining exposed areas. Showed in Fig. 6 pine still absorbed water at this moment.

On spruce the highest point of emission was on a lower lever at the tangential latewood (Ar6 – Ar3) than in pine showed in Fig. 5. The temperature decreased parallelly in every area with differences of 0.15 °C between tangential early- and latewood and 0.40°C between radial and tangential earlywood. Pine decreased the heat of sorption faster and was 1.5°C colder in Ar4 whereas Spruce in Ar4 was only 1.3°C colder after 30 minutes.

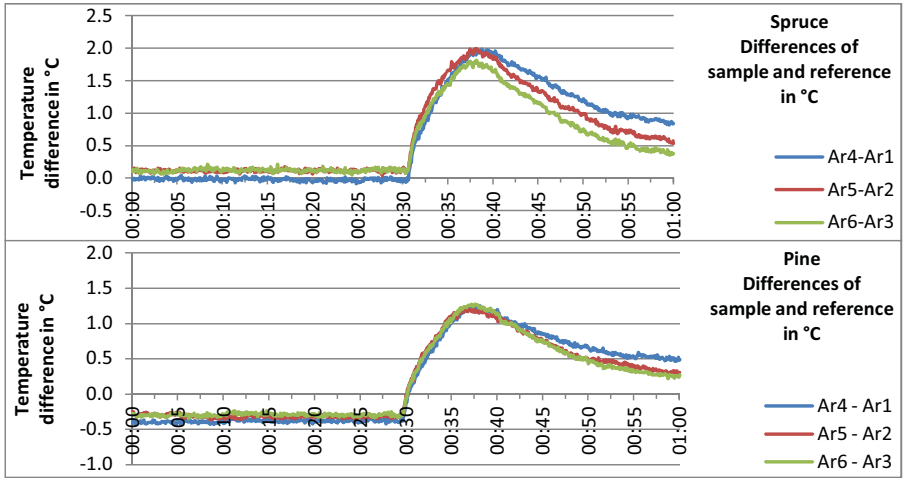


Fig. 5: Temperature difference between areas of sample and reference sample. The decrease is the quotient of sensible heat emission and decreasing values of RH and room temperature in the climatic chamber. The decline of radial earlywood is slower.

Spruce absorbed moisture faster and at a higher rate than pine. The curve in Fig. 6 showed a parabolic character and was constant after 60 minutes. Spruce absorbed 9 g water in 20 minutes. The high gradient at RH load obtained the sorption speed which was responsible for the higher heat of sorption shown in Fig 5.

The water content of pine rose slightly and not linearly at the first 4 minutes. After reaching RH 90% at 00:37 the uptake behaviour was almost linear with an uptake of 0.3 g/min. The highest average weight gain of 9.0 g of pine was reached after 70 minutes remaining with low fluctuation until next climate.

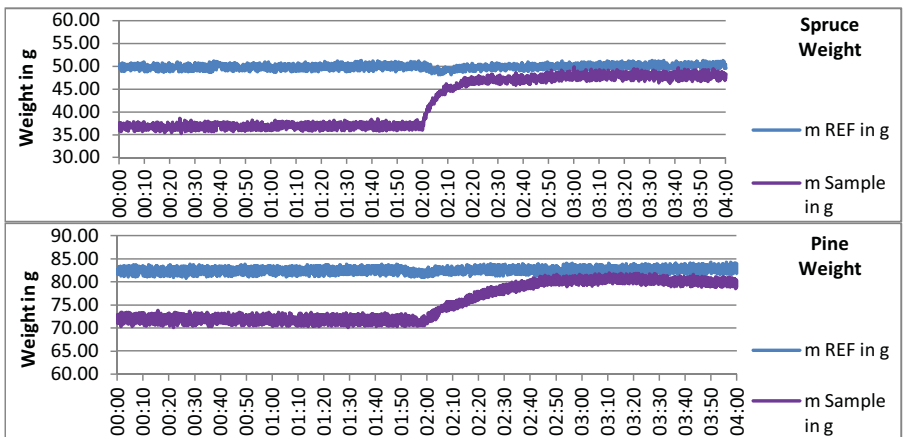


Fig.6: Measured weight of reference and sample in 4h

## DISCUSSION AND CONCLUSION

Latent heat exchange occurred on both species. The surface heating up to 2°C of spruce provides an energysaving potential in similar indoor conditions. Surface temperature compared to the weight gain showed in Fig. 4, unfolds that heat correlates with moisture uptake. The latent heat exchange provides energy in indoor areas for hours after moistening. Thus untreated wood has energy saving potential in every kind of interior application.

Thermography provides an adequate method for measuring temperature differences due to moistening of wood. The comparison of reference samples and samples provided sorption and heat characteristics in correlation with climate with the suitable accuracy for closer examination. The thermal fluctuation of the climate chamber did not cause indiscrepancies.

The periodical recording and reasonable accuracy provides details for future work. Still there are several unknown factors like how MC, water transmission and thermal diffusion influence the surface temperature. Furthermore the amount of energysaving is unknown and needs to be examined closer. The experiment showed that spruce and pine emit heat with same trends during the RH raise or decrease, but differ in temperature amount and rate. The sorption of spruce was faster and provided more heat than pine. The size of pores and the amount of hydrophobic extractives play a significant role for the heat of sorption which will be examined closer to define the suitability of species.

On the long-term the WEEE project will provide a basement for strategies to use wood specifically as a supplement energy source. Future studies will examine wood species at varying indoor climates; their effect on the energy balance and the correlation between latent heat exchange and wood properties. The next experiment will be conducted in test houses of cross laminated timber. The results will be validated in WUFI®Plus in order to be able to calculate the practical effect which are of interests for the Norwegian wood industry.

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# WEIGHT, VOLUME AND PROPERTIES OF ENERGY WOOD – MEASUREMENTS BASED ON ENERGY WOOD SUPPLY CHAIN

Laurila, J.<sup>4</sup> & Lauhanen, R.<sup>5</sup>

## ABSTRACT

The measurement of the amount of energy wood can be based on volume (m<sup>3</sup>), weight (kg) or energy content (MWh). Physically the measuring can be made in the forest, at the roadside storage site or at the heating plant. In this study the differences, variations and correlations between the different energy wood measurement methods are examined. The whole supply chain from the forest to the heating plant was tracked. The material was collected from young stands and it was based on about 14,300 m<sup>3</sup> solid (12.7 million kg) energy wood from 75 worksites. There was a strong correlation between the values given by the different measurement methods throughout this study although a remarkable weight loss (37 %) between the forest and the heating plant was observed. Moreover, the solid volume factor 0.49 between the chips loose volume and the calculated solid volume, based on the crane scale, was higher than the 0.40 factor that is normally used in practice. The average moisture content measured at the heating plant was 43 %. The original research article: "Laurila, J. & Lauhanen, R. 2012. Weight and volume of small-sized whole trees at different phases of the supply chain" is in print: "Scandinavian Journal of Forest Research, 2012; 27: 46-55".

Key words: crane scale, energy wood, measurement, moisture content, volume, weight, whole tree

## INTRODUCTION

There are many problems, when measuring biomass (Rosillo-Calle et al. 2007). The shape of the energy wood is problematic when measuring it. Especially it is difficult to measure small-sized whole trees; which contain a stem, branches and leaves. However,

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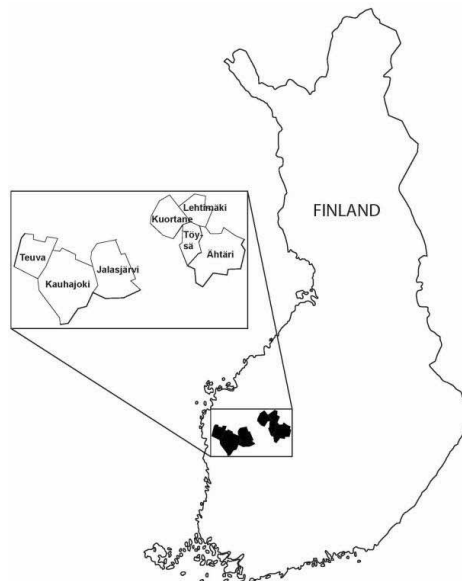
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the yield of wood chips increases by 15-35 % when harvesting whole-trees compared to delimbed stems (Hakkila 2001). The amount of energy wood can be measured based on weight (kg), volume (m<sup>3</sup>) or energy content (MWh). The conversion from one unit to another unit can be unreliable and inaccurate. Moreover the measurement method used can be imprecise. Even the size of the energy wood batch affects the measuring accuracy. In addition, the change in moisture content has made the situation even more complex. The moisture content should be known when the result is based on fresh or dry weight (Hakkila & Parikka 2002). The moisture content range, in Finland, of freshly-felled small-sized Scots pine and Norway spruce trees varies between 50 - 60 % and birch trees 40 - 50 % (Hakkila 1989). The aim of this study was to examine measurement results and weight loss at various stages in the energy wood supply chain based on the wood's weight and volume.

## MATERIAL AND METHODS

The data was collected from energy wood thinning sites (breast height diameter of 8 - 16 cm) between November 2004 and October 2009. The sites were located in seven municipalities in Western Finland (Fig. 1). The total number of energy wood thinning sites was 75. The energy wood was harvested mechanically using the whole tree method. The study data was collected from forest organisations, energy organisations, heating entrepreneurs, machine entrepreneurs, individual forest owners and the authors own measurements.



**Fig. 1.** Data was collected from seven municipalities in Western Finland.

The first measuring of the energy wood's fresh weight happened at the forest work sites using a crane scale. The crane scale weight results (kg) were converted to volume ( $\text{m}^3$ ) by using the fresh density number from the official guide for the measurement of energy wood (Lindblad et al. 2008). The frame volume of the energy wood stacks was measured at the roadside storage sites.

The results from the heating plants are based on declarations from the heating entrepreneurs or the receiver of the energy wood. The data included: the receiving time, the loose volume of the chips ( $\text{m}^3$ ), the wood weight (kg), the energy content of the chips (MWh), the energy density of the chips ( $\text{MWh}/\text{m}^3$ ) and the moisture content (%) of the wood. The moisture content analysis was based on international Standard ISO 589 "Hard coal - Determination of total moisture". The drying temperature was  $105^\circ\text{C}$  and a drying time of 24 h was used. The measuring of the heating value is based on standard: CEN/TS 14918:2005 Solid Biofuels - Method for the determination of calorific value (CEN/TS 14918:2005).

## RESULTS

### Measurements in the forest

The average area of a forest worksite was 2.9 hectares ( $n = 36$ ) and the average harvested tree stem volume was about 60 litres. The main tree species on 92 % of sites was Scots pine, 3 % were Norway spruce, 3 % were birch and 2 % were other deciduous trees. The average tree height was 12 m and the average time gap between the end of logging and the end of forest haulage was 9 days. The energy wood, at a total of 75 worksites, was weighed by crane scale and the average weight per worksite was 172 tonnes; that being about  $190 \text{ m}^3$  based on the fresh density number (Lindblad et al. 2008). The average volume per hectare was  $59 \text{ m}^3$  solid/ha ( $n = 36$ ).

The frame volume was measured at the roadside storage at an average of 16 days after the end of forest haulage. The average frame volume at the roadside storage sites was  $849 \text{ m}^3$  ( $n = 44$ ) and the average height of the stack was 4.2 m. The average diameter of the base of the tree at the front side of the stack was 10 cm. The solid volume percentage was on average 32 % according to Lindblad et al. (2008). Most of the stacks (63 %) consisted of 85 % Scots pine and 15 % birch. Scots pine was the only tree species in 29 % of stacks and no other stacks of single tree species occurred. Also, most of the stacks (85 %) at the roadside storage sites were covered by waterproof paper.

### Measurements at the heating plants

Energy wood was received at the heating plant after an average period of 11 months of roadside storage. The average loose volume of chips was  $381 \text{ m}^3$  per worksite and the energy density of the chips was  $0.86 \text{ MWh}/\text{m}^3$  loose. The average weight of the energy wood between the forest and the heating plant decreased by 37 % ( $n = 23$ ). The average

moisture content measured at the heating plant was about 43 % (n = 26). There was a difference, over the years, in the moisture content measured where in 2007 the average moisture content was 38 % (n = 3), 2008 it was 45 % (n = 11) and in 2009 it was 42 % (n = 10). The coefficient between the calculated solid volume based on the crane scale and the loose volume of the chips was on average 0.49 with a standard deviation of 0.07. Thus one solid cubic meter of wood was equal to 2.1 loose cubic meters of chips. The range of factor was 0.36-0.68. Moreover, there was a big difference in some cases between the total weight in the forest and at the heating plant (Fig. 2).

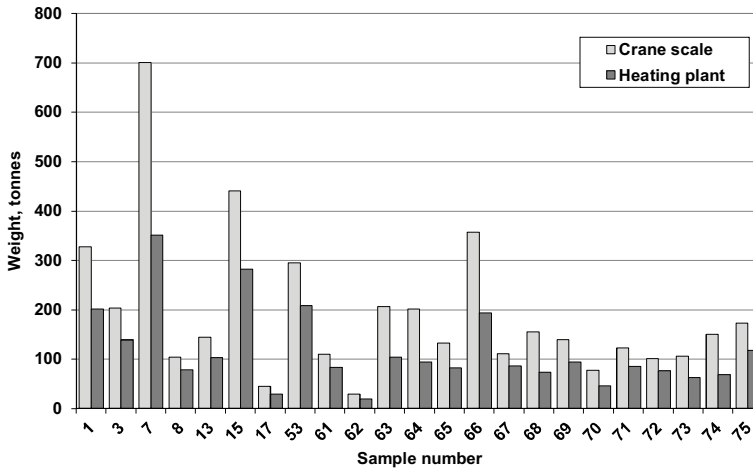


Fig. 2. The weight of the energy wood in the forest and at the heating plant.

## DISCUSSION

The different measurement methods of energy wood based on weight and volume were strongly correlated in this study. However, the weight at the heating plant was only about 63 % of the fresh weight in the forest based on the crane scale. The loss of weight between the forest and the heating plant partly stemmed from the drying of the energy wood at the storage site. In other words a part of the weight loss comes from the loss of water and is desirable.

The average moisture content in this study was 43 % at the heating plant. However, there was a variation (38 - 45 %) over the years. According to Hakkila's (1989) survey the average moisture content of chips at the heating plants (1 - 10 MW) in Finland was 40 % in the winter and less than 35 % in the summer. The survey of Hillebrand and Nurmi (2004) showed that moisture content of small-sized whole trees at the roadside storage was about 40 % after one summer in a good storage place and that covered stacks were dryer (36 %) than uncovered stacks (42 %). However, the average moisture content in this study at the heating plant was about 10 percentage points lower than the

moisture content of freshly-felled small-sized trees. The highest individual loss of weight in this study was 54 % (average 37 %). Thus, the drying of energy wood (water loss) might not be the only explanation for the loss of weight; there has to be also other unknown factors such as dry matter loss.

According to Anerud and Jirjis (2011) the dry matter loss for stump wood can be 8.3 % after 13 months of storage time. A higher dry matter loss of 11.5 % is reported for composite residue logs after a storage period of 8 months (Jirjis and Norden 2005). Similar observations for composite residue logs after 9 months of storage have been reported by Pettersson and Nordfjell (2007). However, even high dry matter loss, if it is realistic, cannot account for the total loss of weight in this study (37 %). It is impossible to know exactly where the disparity came from between the different measurement methods, because there is no absolute value measured in this study. However, it might be possible that the crane scale has a positive systematic measurement error. In the results there was no difference between crane scale types. Loss of foliage and needle mass might be a part of the explanation. Furthermore, a loss of chips is also possible during roadside chipping and transportation.

The coefficient between the calculated solid volume based on the crane scale and the loose volume of chips was quite large. The range of coefficient was 0.36-0.68 and the average was 0.49 with a standard deviation of 0.07. Usually, one loose cubic meters of chips is equivalent to 0.40 (0.38-0.45) solid cubic meter of wood (Kuitto 2005). However, there is quite a large variation between different studies.

The measurement of the amount of energy wood is an important stage for all parties in the energy wood sector. It directly sets the price paid for the energy wood from the viewpoint of the quantity involved. Thus, it is essential that the measurement result is reliable and accurate. Crane scale weighing has been a great improvement in the measurement of energy wood. However, further studies into crane scale weighing and the effect of moisture content on measuring energy wood will be needed.

## ACKNOWLEDGEMENTS

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# EVALUATION OF QUALITATIVE INDICES OF FOREST UNDERBRUSH USED FOR WOOD FUEL PRODUCTION

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## ABSTRACT

The use of plant biomass for energy purposes, including forest residues and underbrush, has many advantages in comparison to fossil fuel. It is considered that underbrush consisting of bushes and imperspective trees is unable to form a new stand. Calorific capacity and moisture content studies of underbrush have not been conducted in Lithuania. The aim of this study was to determine the moisture content, calorific capacity and ash production indices of underbrush (*Corylus avellana* L., *Sorbus aucuparia* L. and *Frangula alnus* Mill.) in separate fractions (stems, branches and leaves). It was estimated that moisture content of underbrush trees is independent of the values of morphometric indices. The ascertained moisture content in leaves of underbrush hazels, rowans and buckthorns is considerably higher than that in stems or branches. However, using underbrush as biofuel, major amount of leaves remains unused. The determined calorific capacity of underbrush (about 18 MJ kg<sup>-1</sup>) and ash production (about 1.1 percent) values of hazel, rowan and buckthorn differ insignificantly from analogous study values of trees and bushes presented in literature. Evaluating underbrush as a source of biofuel, small differences in calorific capacity and ash production among these species are considered insignificant.

Key words: wood fuel, underbrush, moisture content, calorific capacity, ashiness.

## INTRODUCTION

Observing international requirements to reduce the emission of greenhouse effect causing gases (Kyoto Protocol, ratified in 2002), Lithuania's National Energy Strategy (2002) foresees to increase the portion of renewable resources in this primary energy

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balance up to 20 per cent by the year 2025 (Analysis of energy sector tendencies, 2006).

Calorific capacity and moisture content studies of underbrush in Lithuania have not been conducted. The peculiarities of moisture content changes, dependence on brush species, diameter and season have been ascertained.

Some part of underbrush may be used as fuel. Wood fuel, including underbrush, is a source of renewable energy. Qualitative studies of moisture content, calorific capacity and ash production are important for the use of underbrush as biofuel.

One of the most important quality parameters of solid biofuel is its moisture content. At high humidity, fuel is harder to ignite, its calorific value is lower, because some combustion heat is consumed for the evaporation of moisture in the fuel. Burning of wet fuel lowers the combustion temperature, deteriorates heat exchange.

An important parameter of the energy of wood fuel is its calorific capacity. Most often the concept of efficient heat is used, characterizing the amount of heat produced by burning wood without moisture evaporation heat. Calorific capacity of wood containing 30 per cent of moisture is about 5 to 5.3 MWh/t. With increasing moisture content of fuel, its calorific capacity decreases, transportation, storage and burning become more expensive. Oxygen calorimeter is used to determine the calorific value.

Wood burning produces ash. The bigger the amount of ash, the less is the calorific value, however, ashiness of the biomass of all species of wood comprises about 1 per cent. The amount of energy produced by wood biomass is determined mainly by its moisture content, not by the species (Calle, 2007).

## MATERIAL AND METHODS

Studies were conducted in Dubrava Experimental Training Forest Enterprise in Vaišvydava and Šilėnai forest districts. Study objects were selected in stands with prevailing underbrush species of hazel (*Corylus avellana* L.), rowan (*Sorbus aucuparia* L.) or buckthorn (*Frangula alnus* L.) of different density: from very abundant (more than 10000 trees/ha) to sparse ones (1000 and less trees/ha). Underbrush indices were ascertained in the delineated observation plots. Diameters of 1684 underbrush trees were measured at 1.3 and 0.3 m height. Selected and studied were 92 hazel, 63 rowan and 63 buckthorn sample trees.

Humidity calculations of all model trees and their fractions were performed. Typical fragments of model trees were dried.

Upper calorific and ashiness values of the fractions (stems, branches, leaves) of underbrush trees were ascertained by laboratory methods, while lower – by calculations based on the upper calorific value.



## RESULTS AND DISCUSSION

During the study mean moisture content of hazel, rowan and buckthorn fractions (stems, branches, leaves) was measured (Tables 1-3).

**Table 1.** Comparison results of mean values of moisture content in hazel fractions

Fractions of hazel biomass	Mean humidity, percent	p value	Stjudent criterion <i>t</i>	95 % confidence interval of an average	
				lower limit	upper limit
Stems	44,8	0,001	77,8	43,6	45,9
Branches	53,3	0,001	57,1	51,4	55,1
Leaves	62,9	0,001	83,6	61,4	64,3

**Table 2.** Comparison results of mean values of moisture content in rowan fractions

Fractions of rowan biomass	Mean humidity, percent	p value	Stjudent criterion <i>t</i>	95 % confidence interval of an average	
				lower limit	upper limit
Stems	48,4	0,001	135,5	47,7	49,1
Branches	51,5	0,001	98,8	50,4	52,5
Leaves	64,1	0,001	144,4	63,2	65,0

**Table 3.** Comparison results of mean values of moisture content in buckthorn fractions

Fractions of buckthorn biomass	Mean humidity, percent	p value	Stjudent criterion <i>t</i>	95 % confidence interval of an average	
				lower limit	upper limit
Stems	45,4	0,001	84,5	44,3	46,4
Branches	50,6	0,001	88,3	49,5	51,8
Leaves	74,4	0,001	122,8	73,2	75,6

It was found that mean moisture content of the stems of all three species of trees is the lowest in comparison to mean moisture content of branches and leaves.

In the earlier studies it was ascertained that moisture content of wood is the highest in winter, in spring it decreases and in summer it is the lowest. The influence of tree species on moisture content is statistically unreliable (Aleinikovas et al., 2009). Moisture content (45 up to 50 per cent) in Scots pine stems, ascertained by Finnish researchers, was lower than moisture content in branches by 50 – 56 per cent (Alakangas, 2005), as in our case.

Analyzing the relationships of moisture content in different hazel fractions (stems, branches and leaves) with stem diameter at 1.3 m height, it was found that with increasing diameter, moisture content of stems and branches has a tendency to decrease, however, a reliable statistical correlation was not ascertained (Fig. 1 and 2).

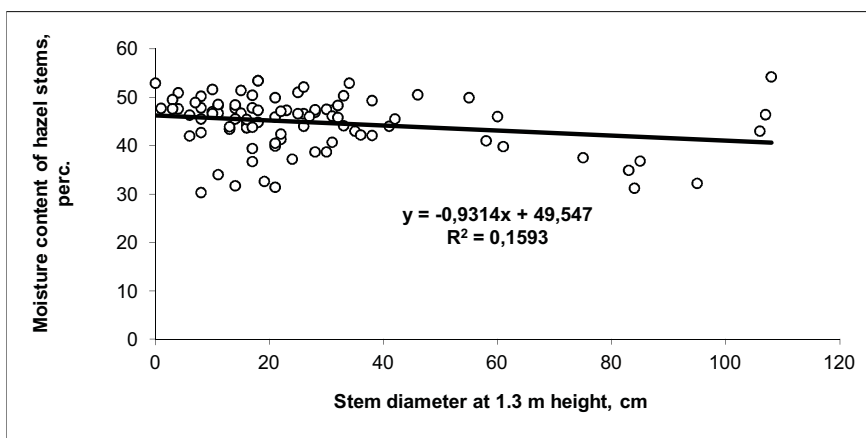


Fig. 1. Dependence of hazel stem moisture content (percent) on stem diameter at 1.3 m height

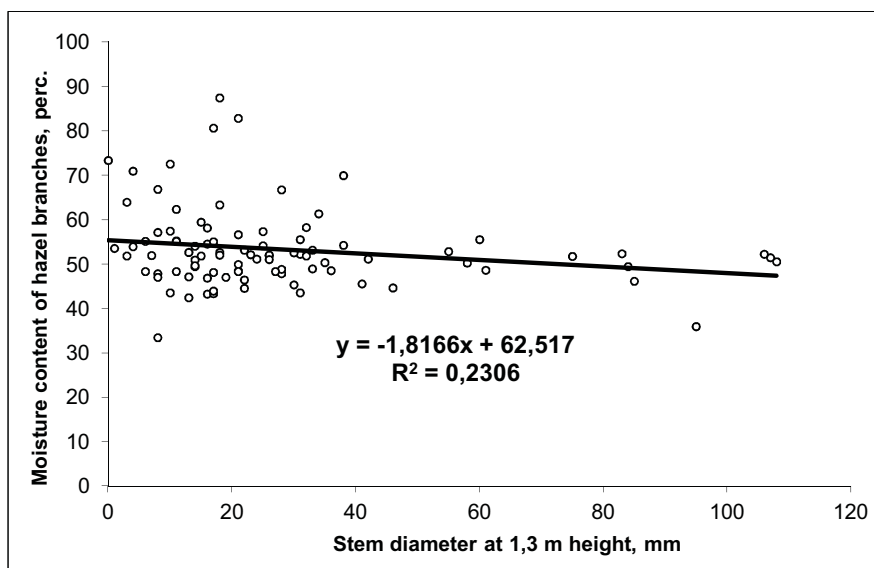


Fig. 2. Dependence of moisture content in hazel branches on stem diameter at 1.3 m height

Russian scientists admit that moisture content of young trees is slightly higher, but with age it decreases.

Correlations of moisture content in different rowan and buckthorn fractions (stems, branches and leaves) with stem diameter at 1.3 m height were also analyzed. Statistically reliable correlations, as in the case with hazel, were not ascertained - the values of determination coefficient were very low.

Upper calorific value and ashiness of dry fuel mass were determined separately for stems, leaves and branches. Experiments were done with 3 replications, mean values of the results are provided in Tables 4 – 6.

**Table 4.** Analysis of hazel calorific values

<b>Fuel marking on the package</b>	Stem of hazel		Branches of hazel		Leaves of hazel	
Ashiness of absolutely dry mass of fuel $A_{s.k.}$ %	0,43		1,22		5,01	
Units	kJ/kg	kcal/kg	kJ/kg	kcal/kg	kJ/kg	kcal/kg
Upper calorific value of dry mass of fuel $Q_v^{sausok.}$	19385	4630	19570	4674	19320	4614
Lower calorific value of dry mass of fuel $Q_a^{sausok.}$	18068	4315	18252	4359	18002	4299

**Table 5.** Analysis of buckthorn calorific values

<b>Fuel marking on the package</b>	Stem of buckthorn		Branches of buckthorn		Leaves of buckthorn	
Ashiness of absolutely dry mass of fuel $A_{s.k.}$ %	0,51		1,4		5,27	
Units	kJ/kg	kcal/kg	kJ/kg	kcal/kg	kJ/kg	kcal/kg
Upper calorific value of dry mass of fuel $Q_v^{sausok.}$	19040	4548	19280	4605	19175	4580
Lower calorific value of dry mass of fuel $Q_a^{sausok.}$	17722	4232	17962	4290	17856	4265

**Table 6.** Analysis of rowan calorific values

<b>Fuel marking on the package</b>	Stem of rowan		Branches of rowan		Leaves of rowan	
Ashiness of absolutely dry mass of fuel $A_{s.k.}$ %	0,32		1,8		3,77	
Units	kJ/kg	kcal/kg	kJ/kg	kcal/kg	kJ/kg	kcal/kg
Upper calorific value of dry mass of fuel $Q_v^{sausok.}$	19201	4586	19516	4661	19530	4664
Lower calorific value of dry mass of fuel $Q_a^{sausok.}$	17883	4271	18198	4346	18211	4349

Lower calorific value of the dry biomass of hazel stem is 18.1 MJ/kg, rowan – 17.9 MJ/kg, buckthorn – 17.7 MJ/kg (Tables 4–6). These indices are slightly smaller than the lower calorific values ascertained during studies in Finland for the dry mass of

birch stem (19.3 MJ/kg), grey alder (19.4 MJ/kg), pine (19.5 MJ/kg), spruce (19.0 MJ/kg), aspen (18.6 MJ/kg) (Nurmi, 1993; 2000).

The determined lower calorific value of the dry mass of hazel leaves is 18.0 MJ/kg, rowan – 18.2 MJ/kg, buckthorn – 17.9 MJ/kg (Tables 4–6). These indices are smaller than lower calorific values of the dry biomass of leaves studied on Finnish tree species: birch (19.4 MJ/kg), grey alder (20.5 MJ/kg), aspen (19.2 MJ/kg), pine needles (21.0 MJ/kg), spruce needles (19.2 MJ/kg) (Nurmi, 1993; 2000).

Calorific capacity of branches of all studied underbrush species was insignificantly higher than that of stems. This was caused by a higher percentage of bark per mass unit than in the case with stems. Calorific capacity of bark is higher than that of wood without bark, even by up to 10-15 per cent (Nurmi, 1993; 2000). The product of wood burning is ash. The higher the amount of ash, the lower the energetic value. Ashiness of hazel, rowan and buckthorn stems comprises about 0.5 per cent, that of branches - between 1-2 per cent. The highest ashiness is characteristic of leaves for all the species, which comprises on an average about 5 per cent. Taking into account that stems make up the bulk of underbrush mass, total ashiness of the mass would be only about 1 per cent, which corresponds to the results of other authors (Calle, 2007).

## CONCLUSIONS

1. Moisture content of underbrush trees is independent of the values of morphometric indices. The ascertained moisture content of leaves (from 62.9 to 74.4 per cent) of underbrush hazels, rowans and buckthorns is considerably higher than that of stems (from 44.8 to 48.4 per cent) or branches (50.6 to 53.3 per cent), however, using underbrush as biofuel, major portion of leaves remains unused.
2. The determined calorific capacity (about 18 MJ/kg) and ashiness (about 1.1 per cent) values of hazel, rowan and buckthorn differ insignificantly from presented in literature analogous study values of trees and bushes. Evaluating underbrush as a source of biofuel, small differences in calorific capacity and ashiness among these species are considered insignificant.

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# RADIOACTIVE CONTAMINATION OF WOOD IN LITHUANIA

Ladygienė, R., Orentienė, A., Pilkytė, L.<sup>1</sup> & Skripkienė, A.

## ABSTRACT

The aim of this paper was to estimate radioactive contamination of wood in Lithuania and evaluate radiation exposure to a population and workers at wood production factories, due to discharge to environment from wood fuel combustion energy power plants, to evaluate effective dose of the population due to living in houses made from radioactively contaminated wood. Radioactive contamination of forests was caused by global fallout from nuclear weapons testing in the atmosphere in the northern hemisphere. Another source of contamination was Chernobyl nuclear accident which resulted in significant contamination of thousands of square kilometers of forested areas with mixture of radionuclides including long lived fission products such as Cs-137 and Sr-90. Part of this radioactivity non-uniformly contaminated territory of Lithuania. The radiological investigation of timber and wood products from different regions of Lithuania was done in 1997-2006. The activity concentration of Cs-137 was very low – ranging from MDA (less than minimum detectable activity) to 4 Bq/kg, the average was  $1.3 \pm 0.3$  Bq/kg. Later, in 2008-2011, Cs-137 and Sr-90 concentrations were measured in wood fuel from the local markets, manufacturers and energy power plants. Activity concentrations of Cs-137 were in range of 0.3-7.0 Bq/kg, Sr-90 – 0.8-6.0 Bq/kg. Average of Cs-137 activity concentration in ash was  $103 \pm 54$  Bq/kg and Sr-90 –  $22 \pm 15$  Bq/kg. Contamination of timber and wood fuel products imported from Ukraine and Belarus were also investigated. Measured activity concentrations were higher in comparison with Lithuanian wood.

Measurements of activity concentration of Cs-137 were performed by gamma spectrometry using IEC 1452:1995 method. Sr-90 activity concentration in the wood was determined after radiochemical separation by counting the Cherenkov radiation of high energetic particles (2.27MeV) radiated by Y-90 in a liquid scintillation counter Quantulus.

Exposure of the population due to contaminated wood depends on activity concentrations of radionuclides in wood and different application of wood products. Modelling of gamma dose rate due to wood contaminated with Cs-137 was performed

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using Visplan-3D ALARA suite 4.0 planning tool and the effective dose to the population was estimated.

*Key words:* wood radioactive contamination, Cs-137, Sr-90, activity concentration, exposure, effective dose

## INTRODUCTION

Global radioactive fallout after nuclear weapons test in open atmosphere and fallout after Chernobyl nuclear accident contaminated Lithuanian forest areas as environment with mixture of radionuclides including long lived fission products such as Cs-137 and Sr-90. Recently short lived radionuclides already decayed. Contamination with Cs-137 and Sr-90 declines slowly. After Chernobyl nuclear accident due to sunny weather when radioactive cloud passed Lithuanian territory (southern and western regions), radioactive pollution fell down in dust form and not heavily contaminated Lithuania [1]. After the accident, the fallout resulted in contamination with Cs-137 up to 18,5 kBq/m<sup>2</sup> and the highest contaminated areas were in some places close to the sea in the western part of Lithuania where contamination was approximately 19,7 kBq/m<sup>2</sup> [2].

Timber and wood products can be used in a variety of ways, in both industrial and domestic situations. Industrial uses are: processing of raw timber in a sawmill; raw timber and /or sawn wood in construction; sawn wood in furniture manufacturing; raw timber and wood pulp in paper and cardboard manufacture; waste wood and bark chips in energy power plants. Contaminated wood can cause the exposure to workers and population. It is important to evaluate effective dose to the population and workers in different situations. Population can get exposure in situations: wood buildings, flooring, furniture, general construction, domestic fuel. For effective dose evaluation investigation of radioactive contamination of wood must be done. It means activity concentration of Cs-137 and Sr-90 must be measured.

Results of investigation of radioactive contamination in Lithuanian wood and wood imported in Lithuania, evaluation of effective dose to the population due to different exposure pathways are presented in this paper.

## METHODS

During the period 1997-2006 different types of sampling of timber and wood products were used. Samples were delivered for analysis by exporting companies or samples were taken from wood-processing factories by staff of RPC etc. The activity concentration of radionuclides in the analyzed samples refers to dry weight. Results were analyzed and included into data base.

Cs-137 and Sr-90 are fission products. Cs-137 is beta and gamma emitter. The determination of activity concentration of Cs-137 in the samples of wood products and

ash were done using method IEC 1452:1995. Two gamma spectrometers with high-purity germanium detectors were used. Relative efficiencies of the detectors were 27.3%, and 25%. The spectra were analyzed using spectra analysis program Genie2000 version G2K-CPCE1 V3.1A. Efficiency of detectors was calibrated using mathematical calibration with program LabSOCS in energy range 60-2000 keV for particular measurement geometries and densities of wood and ash.

Sr-90 is beta emitter. Sr-90 activity concentration in the wood and ash was determined after radiochemical separation by counting the Cherenkov radiation of high energetic particles (2.27MeV) radiated by Y-90 in a liquid scintillation counter Quantulus. Both of methods are accredited according requirements of ISO standard 17025:2005.

2008-2009 samples of wood fuel products were collected from the local markets, manufacturers. 2010-2011 wood fuel products were collected when project "Assessment of radioactive contamination of wood and exposure to population" was implemented. Created questionnaire helped to get information from energy power plants about kind and origin of wood fuel, annual amount of wood fuel, amount of ash and disposal of ash treatment of ash. Answers were got from 32 energy power plants. The ash/dry ratio was determined during combustion process and calculated as a ratio of ash weight and weight of dry wood fuel taken for burning at less than 400°C. At this temperature Cs-137 and Sr-90 do not evaporate and concentrate in ash.

Calculations of gamma dose rate due to Cs-137 in wood building materials were performed by Visiplan 3D ALARA suite 4.0 planning tool which allows to describe sources of any geometry and with any gamma radionuclide composition and to perform calculations of dose rate distribution around these sources. Contaminated wood used like building material gives external exposure indoors.

Effective dose to the population was evaluated using calculated gamma dose rate, occupancy factor (time spent indoors) 0.8, conversion coefficient from gamma dose in air to effective dose 0.7 Sv/Gy.

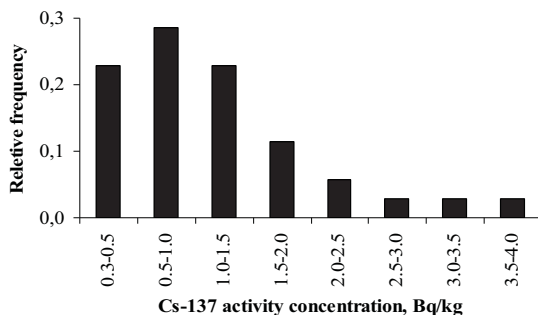
## RESULTS AND DISCUSSIONS

### Contamination of Lithuanian wood

Wood samples investigated by gamma spectrometry were: wooden board, chipboard, parquet, wood fiber, plywood, wooden door, timber. Types of investigated wood were: fir, pine, birch, oak, alder and asp. During production process of wood items Cs-137 activity concentration in wood remains the same.

Cs-137 activity concentration in different kinds of wood products was in range from MDA (less than minimum detectable activity) to  $4 \pm 1$  Bq/kg, average –  $1.3 \pm 0.3$  Bq/kg (95 % confidence) [4], median – 1.0 Bq/kg. Distribution of Cs-137 activity concentration in the samples of Lithuanian wood and wood products is given in Fig. 1.





**Fig. 1** Distribution of Cs-137 activity concentration in the samples of Lithuanian wood

Cs-137 activity concentration in Lithuanian wood is very low. There was very low activity concentration in wood and insufficient number of samples to find statistically significant difference between contamination of wood from different Lithuania regions and between different types of wood. Evaluation assessment efficiency doses to workers is not needed when Cs-137 activity concentration in wood do not exceed exemption level (10000 Bq/kg).

Samples of imported wood from Belarus and Ukraine were also investigated by gamma spectrometry, but there were not many samples. Measured Cs-137 concentration was found up to  $406 \pm 20$  Bq/kg. A few samples of Siberian larch from Russia were investigated. Cs-137 concentration was very low –  $0.4 \pm 0.2$  Bq/kg.

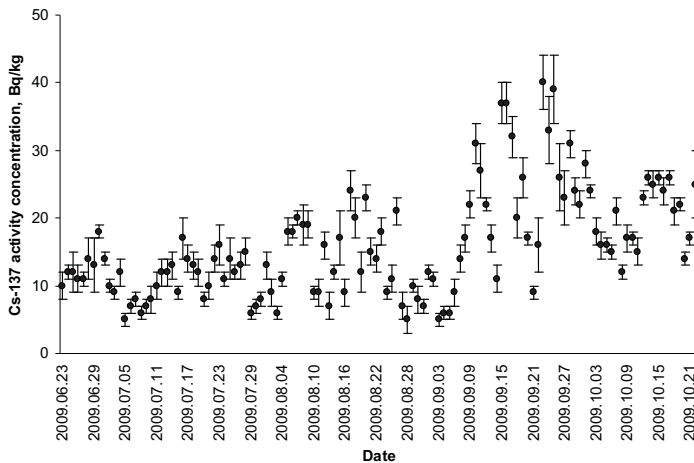
Another way to identify contamination in wood is measurements in living environment. Measurements of gamma dose rate of wooden furniture, wood trim and wooden board were done in living houses. Dose rate did not exceed background. Dose rate indoors were measured in 600 hundred of wooden dwellings built 1915-2005 in whole Lithuania. Increase of dose rate was not identified. It means dwellings were built from non or very little contaminated wood and fallout after Chernobyl nuclear accident do not contaminated heavily surface of houses.

### **Contamination of wood fuel**

Wood fuel production is important industrial sector from of view of radiation protection due to possible contamination of wood and Cs-137 and Sr-90 activity concentration in ash. Wood fuel can be produced from Lithuanian wood, imported wood from Belarus, Ukraine and mixture origin of wood. For investigation of wood fuel (briquettes and pellets) 68 samples were taken from producers and markets. The results of radiological investigation of wood fuel products from Lithuanian wood showed that the maximum activity concentration of Cs-137 in the samples of wood pellets and wood briquettes were  $14 \pm 1$  Bq/kg [3]. According to the information from Lithuanian wood fuel producers, the wood pellets can be made of wood with bark or without bark. It is known that Cs-137 activity concentration is higher in the bark than

in the trunk [5]. It follows that the wood fuel that are prepared using only wood trunk have less quantity of Cs-137 compared to those which are made from different parts of trees including bark. It was identified from measurements as well.

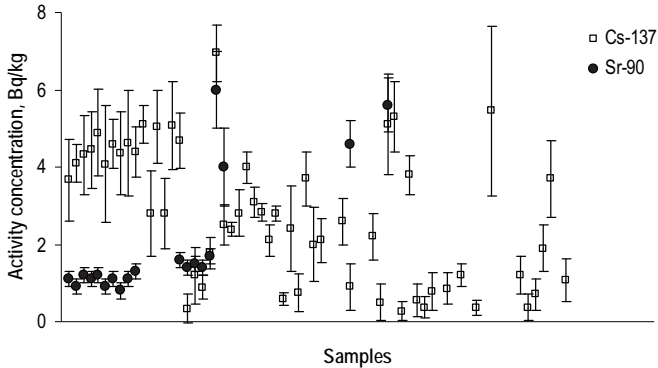
Detail radiological monitoring of Cs-137 in wood pellets was performed at one of the biggest Lithuanian factory (where mixture of wood of Lithuanian origin and Belarus origin were used) taking daily integrated samples (each sample was composed from 24 samples taken on 60 min basis). The measurements of 122 integrated samples were done in the period from June 23 till October 21, 2009. The results of gamma spectrometry analysis showed that Cs-137 activity concentration in the wood pellets was in range from 5 up to 40 Bq/kg (Fig. 2).



**Fig. 2** Cs-137 activity concentration with measurement uncertainty in the samples of wood pellets from investigated factory

Changes of Cs-137 concentration in pellets show that contamination of wood used to produce pellets was different depended on origin of wood.

In 2010-2011 Radiation Protection Centre implemented the project “Assessment of radioactive contamination of wood and exposure to population”. 118 samples of wood fuel and ash were taken from 13 energy power plants. Activity concentrations measured in these samples are given in Fig. 3.



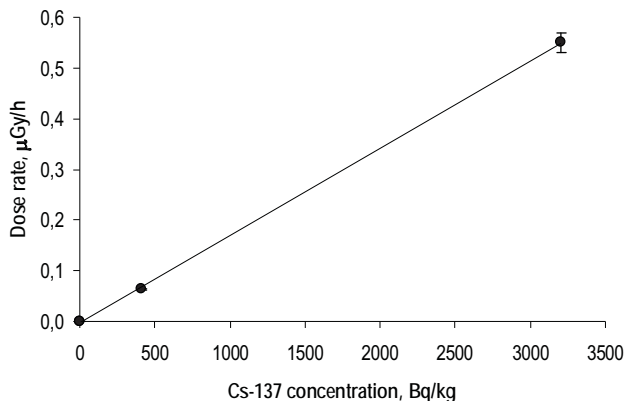
**Fig. 3** Cs-137 and Sr-90 activity concentrations in the samples of Lithuanian wood chips and sawdust

Wood chips and burned ash weight ratio evaluated from questionnaire was from 50 to 370, in average 160. Combustion temperature is 700-1100°C at energy power plants. At such temperature part Cs-137 and Sr-90 evaporate and discharges into the environment. Activity concentrations of radionuclides in wood and in ash, chips/ash ratio, temperature helps estimate what part of activity of radionuclides evaporate. Discharge is one of the pathways of exposure to the population pathways. Dispersion of radionuclides in environment depends on height of a stack. Effective doses due to Cs-137 and Sr-90 discharges from stack was evaluated and do not exceed 10  $\mu$ Sv per year. For calculation conservative approach was used, it means that all activity of Cs-137 and Sr-90 was discharged. Another part of Cs-137 and Sr-90 remains in the ash. Effective dose to the population depends on the way of ash treatment and ash quantity used as fertilizer in agriculture. Level of contamination of soil in such case was estimated. If the maximum permitted quantity of ash, 2.5 tons per hectare per year would be used up to 25 cm in the depth, the ash would contain the maximum measured values of Cs-137 (56 Bq/kg) and Sr-90 (29 Bq/kg), then the radioactive contamination of soil would increase by Cs-137 0.07 Bq/kg, Sr-90 0.03 Bq/kg after a single fertilization. Such contamination will not have significant influence to the exposure of the population.

### **Estimates of population doses due to using wood as a building material**

Wood is used in the construction of houses and other buildings. Wood can be used only for floor, walls or ceiling. For indoor exposure evaluation standard room 4×5×2.8 m<sup>3</sup> is used. Model of this standard room made of wood with wall thickness of 20 cm, density 0.6 g/cm<sup>3</sup> was created in Visiplan-3D environment. Gamma dose rate is caused Bq/kg of Cs-137 concentration and was calculated in the middle of the room at height 1.25 m. Exposure occur from walls, floor and ceiling, provided each of these is constructed of contaminated wood. Each of these surfaces represents a planar radiation source, the significance of which depends on the mass of wood within each surface. For dose rate calculations Cs-137 concentration in wood were used: 1.3 Bq/kg and 4.0 Bq/kg

(average and maximum concentrations found in Lithuanian wood), 406 Bq/kg (concentration found in Belarus wood), 3200 Bq/kg (possible concentration in standard metal wagon loaded wood can pass detector gates on border [3]). Gamma dose rate dependence on Cs-137 activity concentration calculated with software Visiplan-3D is given in Fig. 4.



**Fig. 4** Dose rate in the middle of standard room depends on Cs-137 activity concentration

Dose rate in the standard room made of wood with 4.0 Bq/kg of Cs-137 is 0.6 nGy/h. It cause effective dose for the population 3 μSv per year. This dose is low less than 10 μSv per year (effective dose to the population due to exempted practice should not exceed 10 μSv per year). Effective dose to the population in standard room would be 0.3 mSv per year when Cs-137 concentration is 406 Bq/kg (Belarus origin). Using calculations with Visiplan-3D gamma dose constant for Cs-137 and geometry of standard room made from wood was found  $1 \cdot 10^{-7} \text{ (mGy} \cdot \text{h}^{-1}) / (\text{Bq} \cdot \text{kg}^{-1})$ . It can be used for evaluation of dose rate when activity contamination Cs-137 in wood is known.

## CONCLUSIONS

1. Radioactive contamination of wood from Lithuanian forests is very low and it does not cost any problem from radiation protection point of view and would not lead to increase exposure to the population and workers;
2. Ash of fuel produced from Lithuanian wood used for fertilizers can contaminate soil up to 0.3 Bq/kg for single fertilization;
3. Lithuanian wood used as building material cause the exposure to the population up to 3 μSv per year.

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# IMPACT OF THE SOLVENT ON THE YIELD OF SILVER BIRCH (BETULA PENDULA ROTH.) OUTER BARK EXTRACTIVES

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Jurkane, V.<sup>6</sup> & Tardenaka, A.<sup>7</sup>

## ABSTRACT

Birch in the Northern hemisphere is a very widespread tree genus, which is extensively used in the furniture, pulp and plywood manufacture where, as a by-product, it accumulates birch bark (BB) in the process, which does not find industrial application, and is often burned as fuel. BB comprises about 12.5 wt.% of the tree mass. Outer BB actually consists of a mixture of pentacyclic triterpenes (35–40 wt.%) and biopolyester suberin (45 wt.%). Betulin, betulinic acid and lupeol, representatives of triterpenes, are biologically active substances, whose efficiency can be enhanced by synthetic modification. Betulin in the outer BB powder form is used in folk-medicine as an anti-inflammatory agent. Betulinic acid selectively kills human melanoma cells while leaving healthy cells intact and is found to delay the progression of the HIV 1 infection, which eventually leads to AIDS, by preventing the formation of syncytia. Freshly isolated BB, left over at a plywood factory, was chosen as a representative of industrial waste. Milled dry BB samples were soaked by mixing from time to time in

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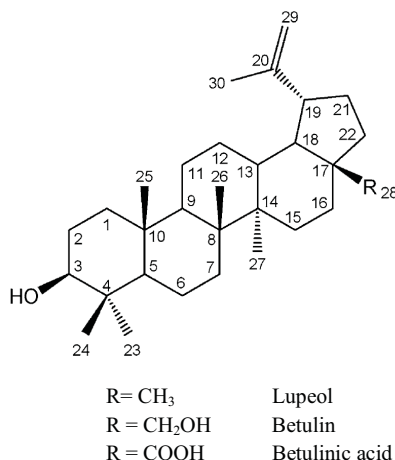
deionized water for 24 h. Outer BB floated on the top of water and this component were collected and dried at room temperature and in a drying cabinet at 50°C up to a moisture content of 1–10 wt.%. Milled outer BB ( $\leq 2$  mm) was pelletized on a laboratory flat die type pelletizer with holes of diameter 6 mm. The temperature equilibrium on the die was approximately 100°C. Extraction of triterpenes was carried out in Soxhlet apparatuses by the selected solvents (ethanol - water mixtures in the range of 75 to 95 vol%, acetone, cyclohexane and heptane) for 1-11 h. The components were identified with gas chromatography/mass spectroscopy by comparison of the spectra with those of the standards. The properties and composition of BB were evaluated to gain information for selecting a technology, convenient for obtaining triterpenes. In the case of the flotation method, the pure outer BB yield was around 40 wt.%. Pelletization increased the outer BB bulk density up to 470 kg/m<sup>3</sup>. The yield of cyclohexane and heptane extractives was considerably lower than that in the case of acetone and ethanol. However, the more selective solubility eliminated the amount of undesirable substances, e.g., polyphenols, tannins, etc. from the total extract.

Key words: Outer birch bark, Flotation, Pelletization, Extraction, Triterpenes

## INTRODUCTION

Birch in the Northern hemisphere is a very widespread tree genus, which is extensively used in the furniture, pulp and plywood production. The main birch species used for veneer log production is silver birch (*Betula pendula* Roth). At the present level of technical development, the extent of a suitable use of the raw material, for example, in the plywood industry is rather high. To produce 1 m<sup>3</sup> of plywood and 0.3 m<sup>3</sup> of wood by-products, 2.75 solid m<sup>3</sup> of birch logs are used. The remainder (veneer shorts, veneer edges, sawdust, cut-off ends, bark and even a part of cores) is turned off to boiler houses. However, this portion of the raw material is still significant and should be diminished. The expensive and valuable natural material must be processed into products of higher added value than firewood.

BB comprises about 12.5 wt.% of the tree mass [7]. 2.0–3.4 wt.% of birch veneer logs' mass is composed of outer BB, and this readily accessible raw material is concentrated already in one place. Outer BB actually consists of a mixture of pentacyclic triterpenes (35–40 wt.%) and biopolyester suberin (45 wt.%). The content of traditional wood components – lignin and carbohydrates is low, i.e., 9 and 6 wt.%, respectively [4, 10]. Betulin, betulinic acid and lupeol (Fig. 1), representatives of triterpenes, are biologically active substances, whose efficiency can be enhanced by synthetic modification. Suberin is a perspective source of higher fatty acids, e.g.,  $\omega$ -hydroxy fatty acids,  $\alpha,\omega$ -dicarboxylic acids and homologous mid-chain dihydroxy or epoxy fatty acids, which are not abundant in nature [4, 10].



**Fig. 1.** Chemical structure of lupeol, betulin and betulinic acid.

Betulin in the outer BB powder form is used in folk-medicine as an anti-inflammatory agent. Its derivative, betulinic acid (Fig. 1), features properties, regulating the development of live cells, which transform the metabolism process in malignant tumour cells in the self-annihilation direction. Betulinic acid selectively kills human melanoma cells while leaving healthy cells intact (it acts against the clinically most important cancer types) and is found to delay the progression of the HIV 1 infection, which eventually leads to AIDS, by preventing the formation of syncytia [1, 6].

The antiviral activity of betulin and betulinic acid was demonstrated against the herpes simplex virus [9]. The great attraction of both those preparations is an extremely low toxicity – LD<sub>50</sub> ≥ 9000 mg/kg [8].

Recently, Chinese scientists have revealed that the inhibition of transcription factors, activating the genes involved in the biosynthesis of cholesterol, fatty acid and triglyceride by betulin, decreases the formation of these lipids. *In vivo* experiments with mice betulin caused weight loss, as well as the decrease of the lipid contents in serum and tissues [11]. Besides the promising therapeutic properties, it is an excellent water-oil system's emulsifier for the production of cosmetic preparations with a positive anti-inflammatory side effect [12].

Though the history of betulin exceeds two centuries, and publications about its production from outer BB and crystallization of betulin date back to the first half of the 19<sup>th</sup> century [5], the interest of researchers has not diminished to this day. The current number of scientific articles and patents about betulin and its derivatives is noteworthy.

Regardless of the constant interest and activities, there are no pharmaceutical preparations elaborated and produced on the basis of betulin and other components of outer BB. We suppose that one of the reasons for such a situation is the lack of a



complex approach to the utilization of BB. Therefore, the objective of our investigation was to evaluate the properties and composition of BB to gain information for selecting a technology, convenient for obtaining triterpenes.

## MATERIALS AND METHODS

### Feedstock

Freshly isolated BB, left over at a plywood factory (BB–PF), was chosen as a representative of industrial waste. Outer bark specimens of growing (crop age 15 years) silver birch (BB–Pend).

BB samples, dried at room temperature (moisture content 2–4 wt.%), were milled in a cutting mill SM 100 (Retsch GmbH & Co) to pass the sieve with holes of diameter 2.00 mm. For analytical pyrolysis needs, the samples were additionally ground to the particle size below 0.25 mm directly prior to analyzing.

### Flotation

Milled dry BB samples were soaked by mixing from time to time in deionized water for 24 h. Outer BB floated on the top of water. Both components were collected and dried at room temperature and in a drying cabinet at 50 °C up to a moisture content of 2–4 wt.%.

### Pelletization

Milled BB–PF ( $\leq 2$  mm) with a moisture content of 2–4 wt.% was pelletized on a laboratory flat die type pelletizer (Amandus Kahl GmbH) with holes of diameter 6 mm. The temperature equilibrium on the die was approximately 100 °C.

### Extraction

Extraction was carried out in Soxhlet apparatuses by the selected solvents (ethanol - water mixtures in the range of 75 to 95 vol%, acetone, cyclohexane and heptane) for 1-11 h. Solvents were evaporated using a rotating vacuum evaporator (Laborota 4003 Control) and dried up to an oven dry state.

### Gas chromatography - mass spectroscopy (GC-MS) analysis

GC-MS analysis was performed on a 30 m  $\times$  0.25  $\mu$ m  $\times$  0.25  $\mu$ m film HP-5MS capillary column (GC-2010 Shimadzu) with high purity helium as a carrier gas at a constant flow rate of 1 mL/min throughout the run. The inlet was operated in a splitless mode at 300 °C. The injection volume was 1  $\mu$ L. The oven temperature was maintained at 160 °C for 1 min and then programmed at 10 °C/min to 300 °C, which was maintained for 15 min. Analysis time was 40 min. The temperature of the mass-

selective detectors was 300 °C. The components were identified by comparison of the spectra with those of the standards and those in the NIST library of spectra.

## RESULTS AND DISCUSSION

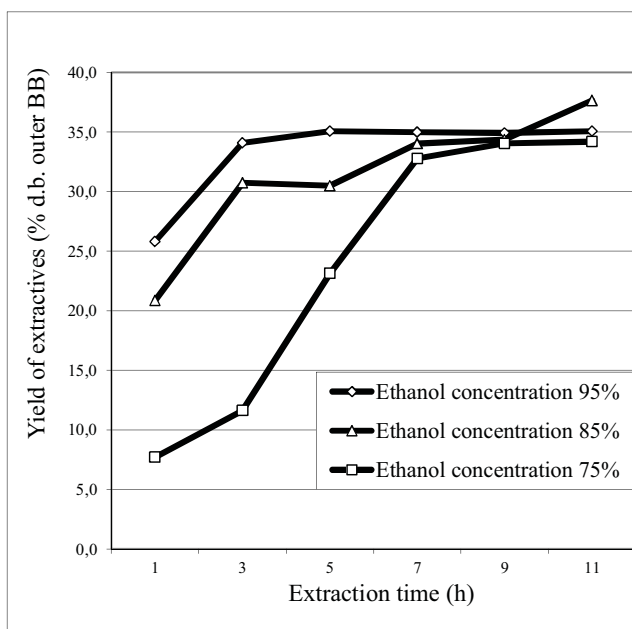
At present and in the immediate future, the main supplier of BB will be plywood factories. After thermal treatment of veneer logs, the bark from the debarker arrives for further processing with an average moisture content of 35–40 wt.% and the bulk mass of 260–270 kg/m<sup>3</sup>. To prepare the raw material for outer BB extraction and qualitative utilization of inner BB, the bark must be dried, crushed and ground in a cutting mill equipped with an appropriate sieve. The ground mass is a mixture of outer and inner BB particles.

To separate the milled outer BB from inner one in laboratory conditions was used flotation method. In the flotation method, the pure outer BB yield was around 40 wt.%.

One of technical deficiencies of outer BB is its low bulk weight: 1 m<sup>3</sup> of dry outer BB from a debarking machine is only 80 kg. After a cutting mill, when its particle size is diminished to 2.0 mm, this index increases up to 210 kg/m<sup>3</sup>. For transportation needs, the only solution is pelletization. Recent technical developments have reached a pelletized outer BB product with a density of 1000–1200 kg/m<sup>3</sup> [3]. Outer BB demonstrated excellent pelletization properties and the resulting outer BB pellets (Ø = 6 mm) had a high bulk density, i. e., 470 kg/m<sup>3</sup>.

Laboratory experiments by making use of Soxhlet apparatuses demonstrated good and almost equal solvent properties of ethanol-water mixtures in the range of 75 to 95 vol%. However, the more diluted ethanol demonstrated a lower intensity of the extraction process (Fig. 2), but the yield of extractives and betulin content in there was similar (Table 1). Only lupeol content showed some differences increasing by the concentration of ethanol.

Betulin is characterized by a low solubility in organic solvents [2]. However, in spite of this, the range of solubility for different solvents is noteworthy, for example, at 15.2 °C, the solubility in acetone is 5.2 g/l, but that in cyclohexane only 0.1 g/l, and at 35.2 °C, it is 13.7 g/l and 0.67 g/l, respectively. In the present study, an attempt was made to elucidate whether the distribution of the extractives' yield from the outer BB of different origin followed the same pattern if the solubility of betulin in the chosen solvents was radically distinctive. The yield of cyclohexane and heptane extractives was considerably lower than that in the case of acetone and ethanol, but the betulin content was the highest. The obtained results are summarized in Table 1.



**Fig. 2.** Effect of the extraction time and concentration of ethanol on the yield of extractives

**Table 1:** Yield and composition of outer BB extractives depending on the solvent type (Soxlet apparatus, 11h)

Solvent	Content of total extractives (% d.b.)	Betulin content (% d.b. extractives)	Lupeol content (% d.b. extractives)
95% ethanol	35.1	61.7	6.0
85% ethanol	37.7	59.6	4.0
75% ethanol	34.2	60.7	3.8
Acetone	39.5	73.6	6.0
Cyclohexane	25.0	86.3	8.5
Heptane	11.5	86.8	4.8

The left over material after the elimination of triterpenes and other soluble matter from outer BB contains suberin as well as somewhat polysaccharides and lignin-like substances. The simplest development of the utilization of this residue is its use as fuel together with fine dispersed inner bark.

## CONCLUSIONS

The excellent pelletization properties of outer BB provide the feasibility to eliminate its low bulk density, which is the main technological and transportation drawback. The best yield obtained in the ethanol-water mixtures was in the range of 75 to 95 vol%, which demonstrated good and almost equal solvent properties; however, the more diluted ethanol demonstrated a lower intensity of the extraction process.

The yield of cyclohexane extractives was considerably lower than that in the case of acetone and ethanol. The change in the pattern of reciprocal solubility may testify the different composition of extractives and a chance to take practical advantage of this phenomenon by the elimination of undesirable substances, e.g., polyphenols, tannins, etc. from the total extract.

## ACKNOWLEDGEMENT

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# THE WATER VAPOUR SORPTION BEHAVIOUR OF MODIFIED WOOD

Hill, C. A. S.<sup>1</sup>

## ABSTRACT

Over the past four years, the Forest Products Research Institute has been conducting wide-ranging investigations into the water sorption properties of wood, natural fibres and cellulose. More recently, this work has been extended to modified wood. The work reported in this paper presents data on the water sorption properties of thermally modified wood, densified wood and a combination of the two. The paper presents new findings concerning the sorption isotherms and sorption kinetics behaviour. The sorption kinetics is analysed in terms of the parallel exponential kinetics (PEK) model which has recently been introduced to the wood science community as a better alternative to the often used Fickian models, at least where cell wall sorption behaviour is considered. The PEK model comprises two exponential sorption kinetic terms which are termed fast and slow sorption processes. The PEK model is then interpreted by using a relaxation-limited kinetics model consisting of two series-coupled Kelvin-Voigt elements. Such a model may be used to provide insights into the sorption process and in particular to the phenomenon of sorption hysteresis. Wood modification has been used as a means of altering the substrate in a controlled manner in order to advance understanding of sorption behaviour. A model for sorption is given in which sorption onto a glassy material below the glass transition temperature can explain hysteresis. Furthermore a link between sorption kinetics and hysteresis is shown in which considerations of molecular relaxation processes within the cell wall can be linked. It will also be shown that the sorption behaviour is governed less by the availability of hydroxyl groups and more by matrix stiffness of the cell wall.

Key words: Thermal modification, water vapour sorption, kinetics, rheology.

## INTRODUCTION

Although the properties of thermally modified wood have been extensively studied, relatively little has been reported on the water vapour sorption isotherms and until very recently (Hill et al. 2012a) the effect of multiple sorption cycles upon the sorption isotherm had not been studied at all. Little is known of the water vapour sorption

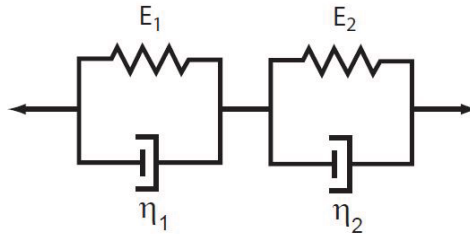
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kinetics properties of thermally modified wood. It is known that densified wood undergoes a relaxation process under humid or wet conditions, caused by the release of internal stresses, one purpose of this investigation was to examine whether there would be any change in the sorption isotherms or sorption kinetics between the first and subsequent sorption cycles. The study was also designed to determine whether this relaxation behaviour characteristic of densified wood could be reduced by thermal modification. It has recently been shown that the water sorption kinetics behaviour of small samples of wood, modified wood, natural fibres and cellulose is non-Fickian and rather obeys a parallel exponential kinetics sorption model (Hill and Xie, 2011, Hill et al. 2010, 2012a, b, Xie et al. 2010, 2011). The PEK model has a double exponential form (Eq.1):

$$MC = MC_0 + MC_1[1 - \exp(-t/t_1)] + MC_2[1 - \exp(-t/t_2)] \quad (1)$$

Where MC is the moisture content at time t of exposure of the sample to a constant RH and  $MC_0$  is the moisture content of the sample at time zero. The sorption kinetic curve is composed of two exponential terms which represent a fast  $\{MC_1[1 - \exp(-t/t_1)]\}$  and a slow  $\{MC_2[1 - \exp(-t/t_2)]\}$  process having characteristic times of  $t_1$  and  $t_2$ , respectively. The terms  $MC_1$  and  $MC_2$  are the moisture contents at infinite time associated with the fast and slow processes, respectively. It has been demonstrated that the PEK model fits the sorption kinetics data for small wood and plant fibre samples with a very high degree of accuracy. It has been argued that the sorption kinetics is determined by the rate of swelling (relaxation) of the cellular matrix. In this model, the two processes are considered to be represented by a pair of series coupled Kelvin-Voigt elements (Figure 1).



**Figure 1: Two series-coupled Kelvin-Voigt elements**

The form of the fast and slow component of the PEK equation is identical with that describing the dynamic response of a Kelvin-Voigt element when subjected to an instantaneous stress increase ( $\sigma_0$ ):

$$\varepsilon = (\sigma_0 / E)[1 - \exp(-t/\varphi)] \quad (2)$$

Where  $\varepsilon$  is the strain at time t, E is the elastic modulus and  $\varphi$  is a time constant which is defined as the ratio  $\eta/E$ , where  $\eta$  is the viscosity. In the present case, there is a change in atmospheric relative humidity (RH) which leads to a response in the wood cell wall.

The maximum swelling pressure ( $\Pi$  – which is here equivalent to  $\sigma_0$ ) that is exerted on an elastic gel when the surrounding water vapour pressure is raised from an initial value  $p_i$  to final value  $p_f$  is given by the following equation:

$$\Pi = - (\rho/M)RT.\ln(p_i/p_f) \quad (3)$$

Where  $\rho$  is the density and  $M$  is the molecular weight of water,  $R$  is the gas constant and  $T$  is the isotherm temperature in kelvin. In the present situation, the strain of the system is assumed to be equivalent to the volume change of the cell wall as a result of water vapour adsorption or desorption. This volume change is assumed to be linearly related to the change in the mass fraction of the water present in the cell wall. The adsorbed water vapour molecules exert a pressure within the cell wall leading to dimensional change, which is considered equivalent to the extension of the spring in the Kelvin-Voigt model.

## MATERIAL AND METHODS

Wood modification was undertaken at Aalto University in Finland. The wood material used in this study was the clear sapwood of kiln dried Scots pine (*Pinus sylvestris* L.). Four specimens were cut from the same board. One specimen was thermally modified, another was surface densified and the third specimen surface densified and subsequently thermally modified. In addition, one control specimen was left untreated. All samples were re-conditioned at 65% RH and 20°C for 3 weeks after treatment. Small wood samples of approximately 5 mg were removed from the surface of the samples for the water vapour sorption analyses using a scalpel. Analysis was performed using a Surface Measurement Systems (London, UK) Intrinsic dynamic vapour sorption apparatus. The sorption cycle started at 0% RH and increased in 5% RH steps to a maximum of 95% RH. The desorption cycle employed a reverse sequence. Three sorption cycles were run, taking approximately nine days. All experimental details have been described previously (e.g. Hill et al. 2012a, b).

## RESULTS AND DISCUSSION

Figure 2 shows the sorption isotherms over three sorption cycles for the unmodified (U), densified (D), thermally modified (TM), and thermally modified densified (TMD) samples. The sorption isotherms for the U wood specimens were found to be essentially reproducible, although there was a small change in hysteresis between cycle 1 and subsequent sorption cycles in the upper part of the hygroscopic range. Densification resulted in a reduction in EMC values for both the adsorption and desorption cycles, but there was a change in the adsorption branch of the isotherm between the first and subsequent cycles. The desorption branch of the isotherm remained the same throughout. The result, therefore, was a significant decrease in the sorption hysteresis between cycles 1 and 2. Closely similar behaviour was observed with the TMD wood specimens, although the combination of the two treatments



resulted in a small reduction in hygroscopicity compared with thermal modification alone. All of the modifications resulted in an increase in hysteresis when compared to the unmodified wood for cycle 1. The phenomenon of sorption hysteresis in wood can be explained by consideration of the sorption process in a glassy polymer matrix (Hill et al. 2010). During the adsorption process microvoids are created within the matrix, whereas these collapse during the desorption process. The rate of response of the matrix to the ingress or egress of sorbent molecules is dependent upon the mobility of the constituent molecules. Below the glass transition temperature ( $T_g$ ) the matrix is unable to respond instantaneously to the movement of the sorbent molecules and consequently the opening and closing of the microvoids in response is delayed somewhat. As a result the adsorption and desorption processes take place in a material that is in different states. It has also recently been argued that the sorption kinetic processes are controlled by molecular relaxation phenomena and that there should be a link between sorption hysteresis and kinetics (Hill and Xie 2011).

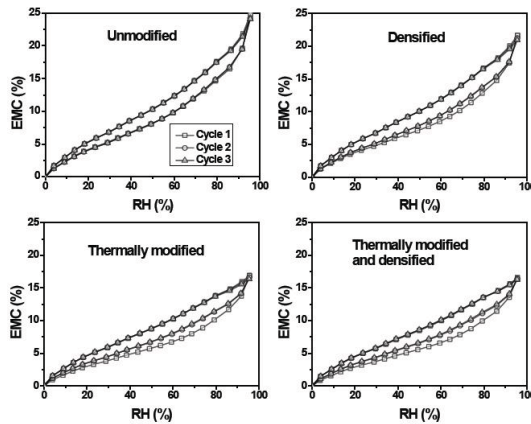


Figure 2: Differences in the sorption isotherms for cycles 1,2 and 3

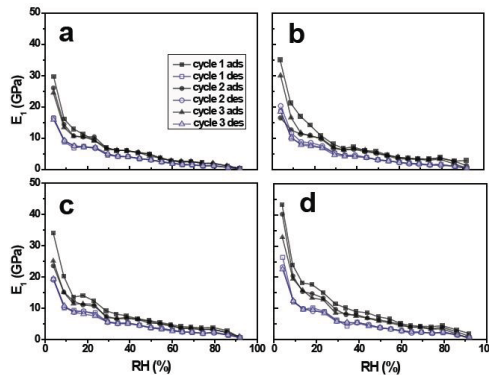


Figure 3: Variation in fast process modulus for unmodified (A), densified (B), thermally modified (C), and thermally modified densified (D) samples

The data obtained from the PEK model can be used to calculate the modulus of elasticity and viscosity of the cell wall. The variation in modulus associated with the fast process for the different wood samples and over the three sorption cycles is shown in Figure 3. A number of features can be observed in these plots. There is a reduction in modulus from 20-40 GPa at low cell wall MC, to somewhere of the order of a few hundred MPa at high cell wall MC. This large decrease can be explained as being due to plasticization of the cell wall matrix macromolecules by sorbed water. However, such a large reduction in modulus is not observed when the mechanical properties of wood are examined at high moisture contents and it is therefore necessary to consider whether this behaviour is realistic. At lower RH the high values of the modulus indicate that there is presumably a contribution from the microfibrils, whereas at the upper end of the hygroscopic range, this contribution is absent and the sorption rate is only apparently determined by plasticised matrix. What is being determined in these experiments is the rate limiting step of the sorption process and it is proposed that polymeric relaxation processes are the ‘bottleneck’. The reduction in modulus may indicate that there is a considerable degree of hydrogen bond breaking and a much greater amount of molecular mobility in the matrix than at lower cell wall moisture contents, leading to a decoupling of any contributions from the microfibrils. An important consideration when discussing these results is that the mechanical properties of the cell wall are being determined by the application of a stress within the cell wall matrix rather than externally. Clearly, at this stage, the approach is speculative and there is a need to undertake independent experiments to verify the conclusions. What form such experiments might take is an open question at this time; it is possible that nanoindentation may be a possible approach, but even here the experiments still use an externally applied stress to determine the material properties. In all cases, the modulus values associated with the fast adsorption process are higher than those found for desorption and there is more variation in the fast process modulus values between sorption cycles.

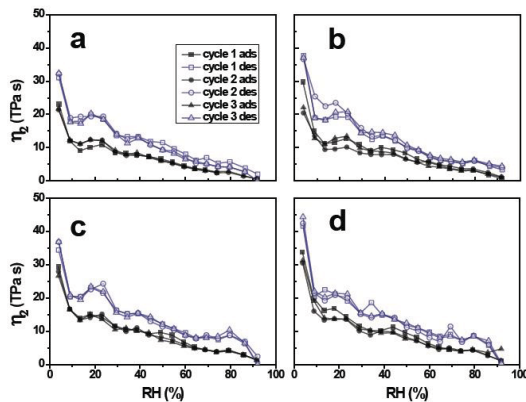


Figure 4: Variation in slow process viscosity for unmodified (A), densified (B), thermally modified (C), and thermally modified densified (D) samples

Wood modification affects the magnitude of the fast modulus associated with adsorption, with the TMD wood exhibiting the highest values. This is also the case for the desorption modulus values, although the differences are smaller. Similar behaviour is found with the variation in slow process modulus. Modification of the wood also affects the adsorption modulus values, which are larger for the TM and TMD samples, but the desorption modulus is scarcely affected. The trends in modulus are consistent with the effects of moderate thermal modification which is often claimed to increase the modulus of elasticity of wood. An increase in modulus is associated with a lower EMC value and the decrease in EMC in the isotherms is divided between the two processes under adsorption conditions, but predominantly with the fast process with desorption. There is a general trend for the modulus values to decrease with each sorption cycle, where such differences occur. This could perhaps be associated with an annealing process taking place within the cell wall matrix. The fast process viscosity is insensitive to wood treatment and also shows little difference between adsorption and desorption as well as between cycles. This is in marked contrast to the behaviour noted with the slow process viscosity (Fig. 4). The viscosity associated with the slow process is always higher under desorption conditions compared to adsorption, whereas the opposite is the case with the fast process. Thermal modification results in an increase in slow process viscosity, an observation consistent with the known effects associated with destruction of the hemicelluloses due to thermal modification.

## CONCLUSIONS

Densification, thermal modification and a combination of those treatments reduce the hygroscopicity of wood, but all result in an increase in the hysteresis between the adsorption and desorption branches of the isotherm for the first sorption cycle. Changes were noted in the sorption isotherms when the wood samples were subjected to three sorption cycles. With unmodified wood these differences were minor and restricted to the upper part of the hygroscopic range, but with the other wood samples there was a large change recorded in the adsorption branch between the first and subsequent cycles, this resulted in a reduction in hysteresis, in some cases to values lower than that found for the unmodified wood. The sorption kinetics behaviour was analysed using the parallel exponential kinetics model and the parameters from the curve fitting procedure were used as input into Kelvin-Voigt series-coupled elements, thus allowing calculations to be made for the moduli and viscosities associated with the fast and slow sorption processes. The moduli associated with the sorption processes are affected by wood treatments, with the modulus increasing in association with the thermal modifications especially. The fast process viscosity is little affected and the main changes due to wood modification are observed with the slow process viscosity.

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# ELECTRIC MOISTURE CONTENT MEASUREMENTS IN THE FIELD TO EXAMINE THE PERFORMANCE OF WOOD BASED MATERIALS

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## ABSTRACT

A system for continuous measurements and long-term recording of wood moisture content has been developed to be implemented in durability field tests. Based on the electrical resistance of wood measurements were conducted using conductively glued electrodes and miniature data logger. Material-specific resistance characteristics were determined for a wide range of native timber species, thermally and chemically modified timbers as well as for timber impregnated with various wood preservatives. The system was thus calibrated in a range between 15 and 50 % wood moisture content. The electrical conductivity was significantly affected by the wood species, the type and intensity of cell wall modification and preservative impregnation respectively. However, moisture content measurements were possible for all materials in question with an overall satisfying accuracy for moisture contents above 15%. The measurement system has been applied to estimate the moisture load under different test conditions, the moisture performance of various wood-based materials, as well as the test-site specific severity of exposure. The system turned out to be a useful tool for determining the time of wetness that occurred on tested wood-based materials under outdoor exposure. However, the results pointed on the need to determine resistance characteristics for every single material under test to assure high reliability. Some limitations need to be considered when wood is treated to varying extents.

Key words: decay, modified timber, resistance, service life prediction, wood durability

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## INTRODUCTION

Wood durability is the consequence of wood ingredients having biocidal or inhibiting effects and the moisture performance of wood. Since fungal degradation of wood requires liquid water in the wood to allow transporting fungal enzymes, a risk of decay is given when the wood moisture content (MC) is above fibre saturation. Secondly, wood temperature has a distinct influence since it determines all bio-chemical reactions. The moisture performance of wood and timber structures is affected by its anatomical structure, the content and type of hydrophobic ingredients and finally the exposure and design of a construction. To characterize the moisture performance of wood and to quantify the effect of moisture loads on the resulting durability it is unavoidable to measure wood MC in the field as well as on real structures in service.

Generally there are different possibilities for measuring and recording wood MC continuously: Direct measurements can be conducted using load cells, e.g. described by Van den Bulcke et al. (2009), which is very precise and allows determining the global MC of a wooden specimen, but not the particular MC at a certain position. Furthermore the method is sensitive to wind loads and the size of test objects is limited. Capacitive MC measurements suffer from a strong relationship between permittivity and wood density, which is often unknown and varies within a test object. Hygroscopic MC measurements are based on the relationship between relative humidity RH, temperature and equilibrium wood MC. From temperature and humidity in a bore hole one can calculate the respective MC (e.g. Evans 2004). However, this method is only applicable for MCs below fibre saturation.

Finally, electrical resistance measurements provide information about wood MC and are quite often used for continuous outdoor measurements and recordings. A system for long-term data logging on timber structures and field trials has been developed by Brischke et al. (2008). It consisted of a 2k-epoxy resin, serving for the isolating glue and also as conductive glue (when mixed with graphite powder and ethanol) in combination with a partly isolated stainless steel cable, acting as both, electrode and cable. This system was further tested in combination with mobile mini data loggers. However, since the electrical conductivity depends on wood species and wood temperature, a calibration is needed for every single material under test. Earlier results showed that measurements with sufficient accuracy were possible in a range between 15 and 50% wood MC for different native softwoods (Brischke et al. 2008). However, cell wall modification as well as the impregnation with preservatives is expected to have a significant impact on electrical conductivity of wood, as for instance reported by Smith et al. (2007).

This study aimed therefore on the determination of material-specific resistance characteristics of native soft- and hardwoods, thermally and chemically modified timber as well as timber impregnated with different types of wood preservatives. Examples of use will be shown to demonstrate the applicability of the system.

## MATERIAL AND METHODS

### Wood materials

Resistance characteristics were determined for a wide range of materials, which were studied in terms of durability field tests and moisture monitoring of timber structures. As summarized in Table 1 native, differently modified and preservative treated timbers were included in the tests.

### Calibration of the measurement system

Data logger (Materialfox Mini, Scantronik Mugrauer) were used for resistance-based measurements. The memory capacity was 16 000. The data logger were equipped with three ports. The measuring ranged from  $2 \cdot 10^4 \Omega$  to  $5 \cdot 10^8 \Omega$ . A sampling interval of 30 s was chosen for the calibration experiments.

Table 1. **Materials used for determination of resistance characteristics.**

	<b>Material</b>	<b>Botanical name</b>
Native softwoods	Scots pine heartwood	<i>Pinus sylvestris</i> L.
	Scots pine sapwood	<i>Pinus sylvestris</i> L.
	Scots pine heartwood resinous	<i>Pinus sylvestris</i> L.
	Southern yellow pine sapwood	<i>Pinus</i> spp.
	Radiata pine sapwood	<i>Pinus radiata</i> D. Don.
	Norway spruce	<i>Picea abies</i> Karst.
	European larch heartwood	<i>Larix decidua</i> Mill.
	European larch sapwood	<i>Larix decidua</i> Mill.
	Douglas fir heartwood	<i>Pseudotsuga menziesii</i> Franco
	Douglas fir sapwood	<i>Pseudotsuga menziesii</i> Franco
Western Red Cedar	<i>Thuja plicata</i> Donn ex D. Don	
Native hardwoods	Beech	<i>Fagus sylvatica</i> L.
	English oak heartwood	<i>Quercus robur</i> L.
	English oak sapwood	<i>Quercus robur</i> L.
	European ash	<i>Fraxinus excelsior</i> L.
Modified timbers	Black locust	<i>Robinia pseudoacacia</i> L.
	Furfurylated Southern yellow pine	<i>Pinus</i> spp.
	Acetylated Southern yellow pine	<i>Pinus</i> spp.
	Thermally modified Scots pine	<i>Pinus sylvestris</i> L.
	Oil-heat treated Norway spruce	<i>Picea abies</i> Karst.
Oil-heat treated European ash	<i>Fraxinus excelsior</i> L.	
Preservative treated timbers	CCA 2 kg/m <sup>3</sup>	<i>Pinus sylvestris</i> L.
	CCA 4 kg/m <sup>3</sup>	<i>Pinus sylvestris</i> L.
	CCA 9 kg/m <sup>3</sup>	<i>Pinus sylvestris</i> L.
	ACQ	<i>Pinus sylvestris</i> L.
	ACQ Micronized copper	<i>Pinus sylvestris</i> L.
	Metal-free	<i>Pinus sylvestris</i> L.

The measuring principle was based on the discharge-time-measurement method. First a capacitor was charged through a small ohmic resistance and then discharged through the material to be measured. Based on the time needed for discharging, the resistance

of the material can be calculated. Lithium/thionylchloride batteries (3.6 V; 7.2 Ah) were chosen, which could be used in a temperature range between -55 to +85°C.

For determination of resistance characteristics eight replicate specimens of 20x30x50 (ax.) mm<sup>3</sup> were used for each MC/wood species combination. Two holes of 4 mm diameter were drilled into the specimens with a distance between the centres of 30 mm parallel and 6 mm orthogonal to the grain. To determine the relationship between the electrical resistance and the wood moisture content (MC) gravimetric and electric MC measurements were carried out in comparison at four target MCs (MC = 15, 18, 25 and 50%) and three target temperatures (T = 4, 20, and 36°C).

To achieve the target MCs the specimens were stored in well ventilated miniature climate chambers filled with saturated solutions of NaCl (75% RH; 15% MC), KCl (85% RH; 18% MC), and K<sub>2</sub>SO<sub>4</sub> (98% RH; 25% MC) respectively. To obtain 50% MC the following procedure was applied: Water pressure impregnation, storing in polyethylene (PE) bags for six days at 4°C, afterwards drying down to 50% moisture content at room temperature, subsequent storage in PE bags for another six days at 4°C.

PTFE isolated stainless steel electrodes were driven into the pre-drilled holes. The moisture measurements were conducted successively at the three temperatures with a Materialfox data logger, the mean values of 4-6 recordings were used for plotting the electrical resistance against the gravimetrically measured MC.

## RESULTS AND DISCUSSION

### Resistance characteristics

The resistance characteristics were determined for each material as a function of electrical resistance R and wood temperature T according to the following equation 1:

$$MC(R;T) = a \cdot T + b \cdot EXP((c \cdot T + d) \cdot R) + (e \cdot T + f) + (g \cdot R^2) + (h \cdot T) \quad (1)$$

MC is the wood moisture content in %

R is the electrical resistance in 10lgΩ

T is the wood temperature in °C

a, b, c, d, e, f, g, h, i are material-specific variables

The accuracy of the measurements was sufficiently high for most materials below fibre saturation (Fig. 1). Only the different modified timbers suffered from higher variation, but still a moisture content range was indicated to estimate the level of wetness. As expected the measuring accuracy decreased drastically with MCs above fibre saturation, but the measurement system still allowed a fairly precise measurement for most of the native timbers. Surprisingly, the preservative treated timber showed highest



accuracy over the whole calibration range. In summary, the immanent need for material-specific resistance characteristics to receive highest possible exactness of measurements became obvious.

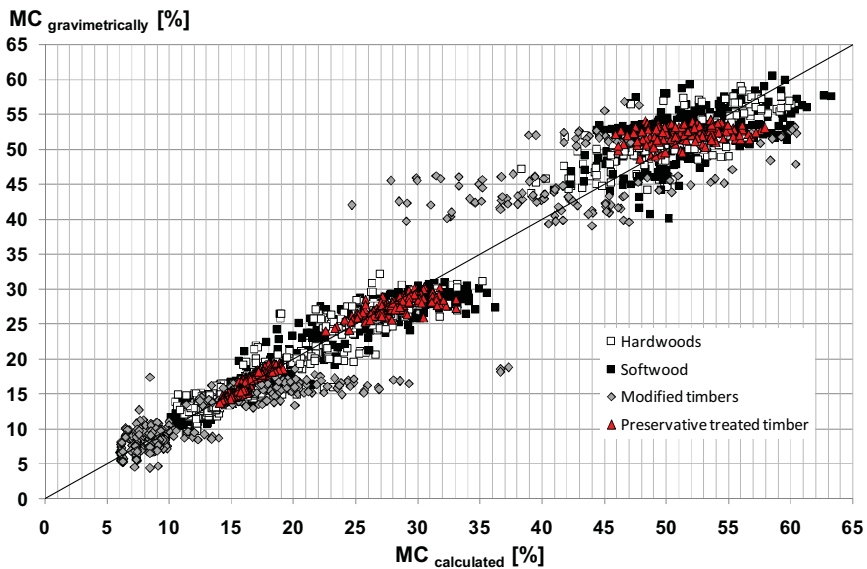


Fig. 1. Calculated MC compared with gravimetrically measured MC.

### Application of the measurement system

The system has been applied within field tests in and above ground (Fig. 2) for moisture monitoring of different materials to estimate moisture loads under different test conditions (field tests and real structures in service), the moisture performance of wood-based materials, as well as the test-site specific severity of exposure. The system turned out to be a useful tool for determining the time of wetness occurring under various exposure situations.

Preliminary results from field tests representing four different exposure situations in and above ground at the test sites Hilo/Hawaii (USA), Borås (Sweden) and Hannover (Germany) were plausible and indicated a reasonable differentiation between test materials. As shown in Fig. 3 differently modified materials suffered from very different periods of wetness. The results obtained from the various exposure situations allow 1. characterizing the moisture performance of the material, and 2. quantifying the occurring dose in terms of moisture and temperature loads to be used for the development of performance models (e.g. Frühwald Hansson et al. 2012).



Fig. 2. Left top: Moisture monitoring on a timber bridge. Left bottom: MC recording on sandwich specimens made from modified and preservative treated timber. Right: Instrumented facade deck elements to quantify the effect of climatic changes on the moisture load of timber structures exposed at various locations in Europe

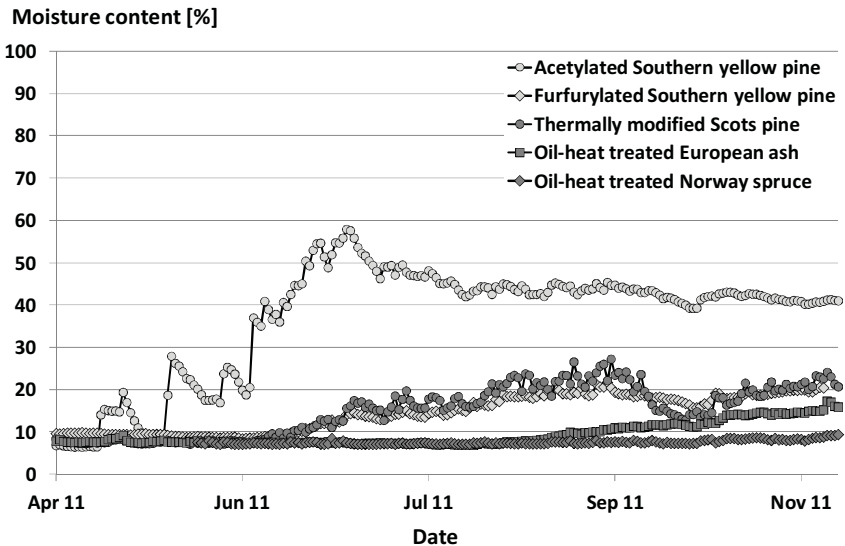


Fig. 3. Moisture course of modified timber exposed in double layer tests in Hannover.

Furthermore, real structures in service have been monitored as shown exemplarily in Fig. 2. A self-supporting timber bridge, where decayed elements and others in question were examined over a period of three years. So-called substitute dowels were used to measure within decayed areas. Electrodes were glued in dowels made from Scots pine sapwood impregnated with tebuconazole solved in white spirit.

## CONCLUSIONS

The results of the study pointed on the possibilities to apply electric wood MC measurements on different variations of wood durability tests and highlighted the strong need to determine material-specific resistance characteristics to obtain highest possible accuracy of the measurement.

## ACKNOWLEDGEMENTS

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# STORAGE PROPERTIES OF TORREFIED WOOD AND CHARCOAL –DECAY RESISTANCE

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## ABSTRACT

Torrefaction is an efficient pre-treatment method that can significantly increase the energy density, overall quality, and handling characteristics of biofuel. Roasted in relatively mild temperatures, usually in 200–300°C, in absence of oxygen and in atmospheric pressure, the properties of feedstock improve and approach those of coal. Torrefied wood can be used in co-combustion with coal as well as for production of syngas and bio-oil. Still, there are a number of questions that are related to the use of torrefied wood in power plants, such as the storage properties. To quantify the outside storage properties of torrefied wood, this article presents preliminary results that focus on the resistance to decay.

Keywords: Torrefaction, moisture, decay, mass loss

## INTRODUCTION

The global need to decrease fossil fuel emissions and the increasing demand for energy has created an interest towards renewable energy sources. Biomass is the primary source of renewable carbon that can be utilized in production of biochemicals as well as liquids and solid biofuels (Medic et al. 2012). Despite its potentials, biomass has its problems. Low bulk density, high moisture content, hydrophilic nature, biodegradation, low energy density, and heterogeneity make biomass utilization challenging (Medic et al. 2012, Arias et al. 2008, Obernberger and Thek 2010, van der Stelt et al. 2011). To make the feedstock and therefore the product more uniform, torrefaction can be used. After treatment in moderate temperatures the resulting product is more hydrophobic, brittle and energy dense (e.g. Bergman et al. 2005, Acharjee et al. 2011). The most reactive parts of biomass, i.e. the hemicelluloses, undergo decomposition so that a carbonaceous solid, a liquid fraction and volatiles are formed (Bourgois and Guyonnet 1988, Prins et al. 2006b).

The storage of biofuels is challenging due to the tendency to degrade and absorb moisture. Wood chips are often stored uncovered outdoors, because it is an easy and

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cheap option. However, the decay resistance of untreated wood is poor and the piles suffer from mass loss and deterioration of quality through microbial activity (Bergman and Nilsson 1979). According to Bergman et al. (2005), torrefied wood obtains only 1–6 % of moisture after treatment, depending on the conditions. Hydrophobic traits appear in the dehydration reactions where the capability of biomass to form hydrogen bonds decreases due to the destruction of many OH groups and formation of unsaturated, non-polar structures (Bergman et al. 2005). Since decaying agents require minimum 18 % moisture content (Lindgren and Eslyn 1961), torrefied wood should therefore be resistant to biodegradation. In addition, the destruction of sugars in hemicellulose makes treated wood less suitable for fungi. However, there are several bacteria and fungi that can also utilize and decompose much dryer material, such as brown coal, lignite and even hard coal (Cohen and Gabriele 1982, Hofrichter and Fakoussa 2001). Mould and staining fungi will not significantly affect the properties of wood but are known to cause respiratory problems for workers and pose a threat for working safety (Diehl 1998). Many white-rot and brown-rot causing agents are effective decomposers and can lead to severe mass losses (Levin and Castro 1998).

In Finland, wood and logging residues are the most important carbon neutral biofuel and their use could easily be increased (Laitila et al. 2008). The logging of mature spruce forests produces a notable amount of crowns and stumps for energy use and in the near future, the thinning potential in young pine and birch stands is also going to increase (Laitila et al. 2008). As a major part of the logistic chain, storage issues should be taken into careful consideration to prevent losses of raw material. To study the decay resistance of torrefied spruce and birch wood, an experiment with four different wood utilizing fungi was carried out. The aim of the study was to determine the ability of selected fungi to degrade and utilize wood torrefied in different temperatures in order to conclude the suitability of the raw material for outdoor storage.

## MATERIALS AND METHODS

Blocks of birch (*Betula pubescens*) and spruce (*Picea abies*) wood in approximate sizes on 5x5x5 cm were carbonized in 25 kg batches in four different temperatures: 220°C, 260°C, 300°C, and finally 450°C to acquire charcoal. The blocks were mainly stem wood, with the bark intact. Before carbonisation the samples were dried to an average moisture content of 5–6 % (dry basis) in a cold-air-dryer. To ensure thorough conduction within large blocks, temperature was raised in steps with a holding time of 60 minutes in 110°C, 60 minutes in 170°C and 3 hours in the final temperature. Treatment in 450°C required an extra step in 290°C in order to prevent damage to the reactor. The torrefaction was performed in a pilot plant scale reactor managed by the Kouvola region vocational school.

To test the decay resistance, three different wood decaying fungi and one mould fungus were selected. *Phanerochaete chrysosporium* Burds. is an effective white-rot fungus that is also used in biopulping industry (Blanchette et al. 1992, Fischer et al. 1994) and is even known to degrade coal (Bumpus 1989). *Pycnoporus cinnabarinus* (Jacq.) P.

Karst is widespread in whole Finland and causes white-rot. It is found also on charred wood and in forest fire areas (Niemelä 2005). *Gloeophyllum sepiarium* (Wulfen) P. Karst. is abundant in the whole country. It causes brown-rot and prefers coniferous wood. It is often found in forest residues and chip piles. The genus *Trichoderma* consists of many different species. It is one of the most common molding fungi found in chip piles (Lindgren and Eslyn 1961, Bergman and Nilsson 1967). The strains were obtained from Fungal Biotechnology Culture Collection (FBCC) maintained in the Department of Food and Environmental Sciences, Division of Microbiology, University of Helsinki.

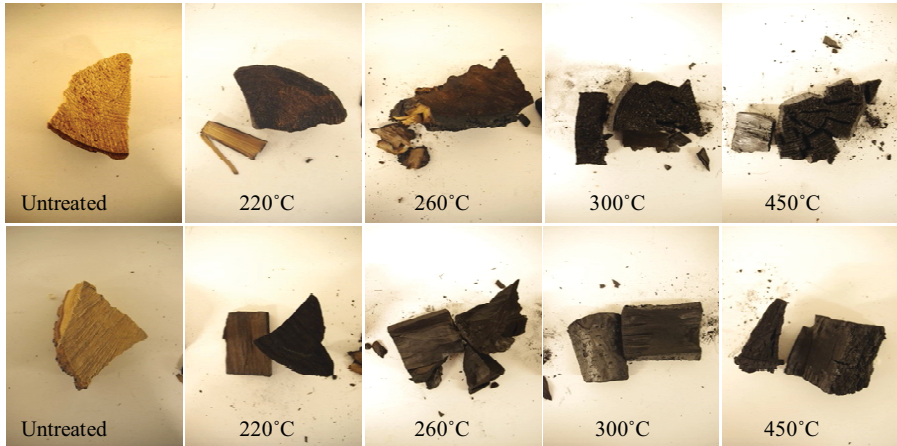
Few blocks of each torrefaction batch were broken into smaller pieces. Samples were taken for C/N-analysis with VarioMAX CN (Elementar Analysensysteme GmbH, Germany). For the decay test, the moisture content of the sample pieces was determined (105°C for 24 hours). The pieces were then sterilized in an autoclave (120°C for 20 minutes) and placed on Petri dishes with a 2 % water-agar medium. The four different fungi were grown on 2 % malt-agar medium for several weeks and small plugs were inoculated on the dishes. Untreated wood was used as reference material. The dishes were incubated in 25°C and 80 % RH for two months. Change in weight was monitored and the growths of the agents were determined by photographing. The first measurements were made after 30 days of incubation and the second measurement after 60 days of incubation. The test was repeated three times. Altogether, 40 dishes were prepared for each repetition, with five treatments for two tree species with four different fungi (5\*2\*4=40).

## RESULTS AND DISCUSSION

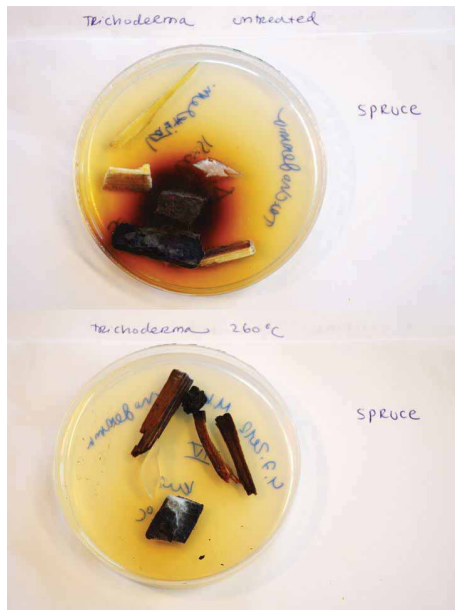
The two tree species reacted to the torrefaction in different ways. The birch blocks were much more charred than the spruce blocks. Already in 260°C the blocks were black, porous and broke easily when under tension, while the spruce blocks were still quite hard and light in colour (Fig. 1). This is most likely due to the different composition of the most reactive component, the hemicellulose. The hemicellulose in deciduous trees is mainly composed of glucuronoxylan, also called xylan, and it is said to be more reactive than the hemicellulose of conifers, which is mainly galactoglucomannan (Prins et al. 2006). Xylan starts to decompose rapidly around 200°C and this leads to a greater weight loss in hardwoods than in softwoods (Prins et al. 2006). The long holding time might also have affected the extensive charring, so two comparative test drives were executed for birch in 250°C and 300°C, with half the residence time. The results from the C/N-analysis comparing the differences in carbon content are not yet available.

After the first measurement of the incubated samples, growths of fungi were clearly visible in all the untreated samples and some of the treated samples. Some of the samples with fungal growth can be seen in Fig. 2. The weight loss was small, but quantifiable in all samples already after 30 days. The weight of the samples included the dish and the water-agar medium. The change in weight in percentages varied

between -0.33 and -1.57 for the spruce samples, with the highest change, surprisingly, in 450°C with *Trichoderma* sp. This is most likely due to the condensation of moisture, which occurred in some dishes, since there was no widespread growth on the samples. For birch samples, the range for change in weight was -0.31–(-2.9) %, with the largest value for untreated birch wood with *P. cinnabarinus*.



**Fig. 1.** Torrefied wood samples, with spruce above and birch below.



**Fig. 2.** Example pair of dishes with spruce wood (untreated and treated in 260°C), showing the growth of *Trichoderma* sp. after 30 days of incubation (coloured areas).



The samples were evaluated visually to determine the growth of the fungi. *Trichoderma* sp. was the most extensive, with small growths even on the samples carbonized in 450°C. *P. cinnabarinus* had small but visible growths on the spruce wood treated in 300°C as well as *G. sepiarium*. *P. chrysosporium* had small growths in spruce wood treated in 450°C and on birch wood treated in 300°C. This shows that in favourable conditions, fungi can utilize torrefied wood and even charcoal to a certain extent. This extent will be determined later with more tests. However, the decay resistance of torrefied wood seems to increase with increasing treatment temperature. Untreated wood suffered the most damage when evaluated visually. Birch seemed to have less fungal growth, most likely because of the more extensive charring during torrefaction. The second evaluation after two months of incubation will give more answers. Also data from C/N-analysis can be obtained after the whole experiment is over.

In actual chip piles that are stored outside, the temperature tends to increase rapidly during the first weeks and in compacted piles it might reach 50 to 60°C (Bergman 1985). Warm conditions favour thermophilic fungi, which were not considered in this study. The self-heating is usually due to respiration of wood cells that remain living some time after the chipping, but with dead cells autoxidative reactions take place (Assarsson et al. 1970). These reactions are exothermic and might also come in question with torrefied material. Thus, self-heating and consequent wetting would promote fungal growths and degradation also when storing torrefied material.

## CONCLUSIONS

Blocks of *Picea abies* and *Betula pubescens* were carbonized in four different temperatures. Samples of each batch were incubated for one month with four different types of fungi. Untreated wood was used as reference. The mass loss and fungal growths were measured by weighing and photographing. After visual assessment it can be concluded that torrefaction increases the decay resistance of wood to a certain extent. The growths were smaller or absent with increasing treatment temperature. More studies will be needed in the future on the self-heating behaviour and the appearance of thermophilic fungi to make viable assumptions on the suitability of torrefied wood for outdoor storage.

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# DURABILITY OF SELECTED LARCH SPECIES AND SCOTS PINE (*PINUS SYLVESTRIS* L.) HEARTWOOD

Pockrandt, M.<sup>1</sup>

## ABSTRACT

Water uptake, in- and above ground durability of larch heartwood of two species (*Larix sibirica* and *Larix decidua*) from two origins (*Larix sib.* from Siberia and Sweden, *Larix dec.* from Sweden) including a comparison to Scots pine sap- and heartwood were studied according to several standard durability tests. The study material was collected and tests started in 1998. The studied larch species and pine heartwood did not show a significant difference with regard to water uptake, while pine sapwood demonstrated significantly higher ability to absorb water. In order to determine the above ground durability of the wood species, an evaluation of the lap joint area was employed. A clear difference between the tested specimens was observed; the Siberian larch from Siberia showed the highest decay resistance while the pine sapwood was non-durable at all. The twelve-year-long exposure in ground contact confirmed the high durability of the Siberian larch wood from Siberia. Differences in the field performance were observed between the two field of exposure, i.e. Simlångsdalen and Ultuna.

Key words: Decay, durability, field testing, Scots pine heartwood, Siberian larch, water uptake

## INTRODUCTION

Durability of wood is an important technological property, which suggests various uses and their service time. An important source for the solid wood industry is the larch wood: approximately 10 species of *Larix* are deciduous conifer trees that are common in northern Eurasia and North America. The species *Larix sibirica* Ledeb. and *Larix decidua* Mill. predominate, and the interest to larch has increased in Finland, Germany and Norway as well (Kewenter 1998). Despite the origin and larch species, larch timber is always marketed in Sweden under the name "Siberian larch". This can be highly misleading for the customers and introduces durability problems during the outdoor exploitation. *Larix decidua* and other species are often used for reforestation in lowlands. From a mechanical point of view, larch wood is valued with good properties

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(bending strength  $\sim 93 \text{ N/mm}^2$ ) and is predominately used for a wide range of applications. Color, which has a red character that is more intensive in the heartwood than in the sapwood, and the texture have also been appreciated. In comparison to the *Larix sibirica*, *L. decidua* grows higher (up to 54 m) and has a larger diameter (up to 2 m). An old problem is still the unclear natural durability of the larch wood. Every wood species has developed to produce and store more or less extractive compounds that can protect the wood. With regard to this, it is already known that larch wood has a high concentration of water soluble extractives (Hakkila and Winter 1973). Heartwood has more stored extractives than sapwood. All heartwood extractives are formed at the sapwood/heartwood border. With a higher age, the concentration of the extractives increases. (Viitanen et al. 1997). The main part of the heartwood extractives consists of arabinogalactan, a water soluble oligomer consisting of the monosaccharides arabinose and galactose. The content of arabinogalactan is determined to be between 5-30% (Côté et al. 1966), and it serves as a nutrient for the fungi. Flavonoids in the heartwood are founded up to 3.5%. Resin- and fatty acids as well as triacylglycerols take only a little part in the extractive content (Viitanen et al. 1997). At least, the role of the different chemical compounds is not clear. A study of G. Koch (2007) shows that there is no clear relation between the raw density and the mass loss caused by decay fungi on larch wood collected from three sites in Siberia. Koch's results show that a wide range of mass loss exists; the highest resistance owns the Siberian larch that is settled in the southern part. There is also no relation between the annual rings and the density. The conclusion of the study is that the natural durability of larch should be dependent on the extractives. A question arises whether the timber from larch species grown at different growing stands (e.g. Siberia and the Nordic countries) have similar properties and behaviour during service. Studies of larch wood decay resistance have been carried out during the sixties by scientists of the USSR Academy of Science, The Russian Institute of Forestry and Wood (Bazhenov and Kharuk 1967, Kharuk 1961) and the Siberian Technological Institute (Shaltyanene 1962). Telegraph poles have been used for the study and they have showed that the average durability of larch wood poles was 19-23 years in the Abakan region and 24 years in the Krasnojarsk region (Bazhenov and Kharuk 1967). The first sign of degradation has appeared after 4 years of use. After 4-15 years of use, 10-20 % of the telegraph poles have been destroyed. However, after 25 years, almost 50 % of the poles have still been in use. As a result of these studies, Shaltyanene (1962) has suggested that the larch is resistant to decay and can be used in ground contact without preservative protection. Comparative data on the resistance of spruce and larch wood poles are shown by Gorshin (1977). The data shows that spruce wood poles have an average durability of 7-8 years while the larch poles serve 14-24 years. Chubinsky et al. (1998) explain the decay resistance of larch by its high density and natural resin content. Data about the high decay resistance of larch wood has also been given by Varfolomeev (1995). Reading the results of these studies, one could easily conclude that larch is a very durable species. Despite this, some drawbacks can hinder the interpretation of the results for usage of larch in Europe. The majority of studies are carried out on Siberian larch grown and tested there. The researchers cannot deny the durability of this species but most of the tests are done in Siberia, a place with rather cold and specific climate particularly in the winter, thus making the conclusions irrelevant for the same species exposed in another

climate. The wide variation of decay resistance within the same wood species has also been observed (Viitanen *et al.* 1997). According to this work, decay resistance depends on both genetical and environmental factors. In Sweden, a preliminary study (Terziev 1998) showed that the European larch (*Larix decidua* Mill.) has significantly higher susceptibility to fungal discoloration than Siberian larch (*Larix sibirica* Ledeb.) and Scots pine. A recent study of Chubinsky (2003) showed that water-soluble extractives of Siberian larch heartwood do not provide any fungi inhibiting effect themselves but correlate best and negatively with the mass losses caused by wood destroying fungi. This is an interesting finding having in mind that the carbohydrates are those compounds easily extracted in water rather than phenolic compounds. It is suggested that Siberian larch heartwood is not more durable than Scots pine heartwood but it seems that the wood has some specific defending mechanism against decay fungi, although it contains significantly more arabinogalactan than Scots pine heartwood, i.e. a carbohydrate which acts as a nutrient for the microorganisms. Other interesting aspects when discussing the durability variations of larch heartwood are the age and density of the material. The composition and the increased amount of extractives with the age can shed light on this difference. The basic density was also found to correlate significantly and negatively with the mass loss. This result is somewhat unexpected and contradicts the findings of similar previous studies (Boutelje and Nilsson, 1985). Since the larch timber is a "new" material in the Nordic countries, there is a scarcity of information concerning its processing and properties. Some data can be found in studies carried out in Russia (Gorshin and Chertsov, 1966, Krechetov 1976, Rasev 1985), but it is often impossible to adjust the old experience to the modern wood technology in the Nordic countries. The objectives of the present study are to compare the durability of Siberian larch grown in Sweden and Siberia, European larch and Scots pine heartwood from Sweden. The study is based on a long-term in- and above ground field tests but a laboratory decay test is included as well. The study is aimed at explaining the nature of the found durability differences.

## MATERIAL AND METHODS

### Material

In order to show the effect of the environment and serve better the needs of the local companies and those importing larch from Russia, larch grown in Sweden and imported from Siberia were compared. The studied species were Siberian larch (*Larix sibirica* Ledeb.) grown in Siberia and Sweden and European larch (*Larix decidua* Mill.) only from Sweden. Sap- and heartwood of Scots pine (*Pinus sylvestris* L.) were used as controls. The trees were selected and cut in 1998. Three logs of each wood species were sawn to 50-mm-thick planks. After drying in room climate, stakes, lap-joint test samples, samples for water absorption and durability tests were cut from the planks. Sapwood was carefully omitted in all larch samples while Scots pine timber was divided to sap- and heartwood samples.

## **Standard ground and above ground field tests**

The durability of larch and pine species was tested in ground contact according to the European standard EN 252. The above ground test was performed according to the standard ENV 12 037 (lap-joint method). For this research, two test fields were chosen namely, Simlångsdalen and Ultuna (both in Sweden). The samples in ground and above ground exposure were set in the fields in 1999.

### **Determination of water absorption/desorption coefficient of the studied species**

Ten stakes were taken from every species to cut samples. Every chosen stake was used to cut 1 stick with a cross section of 25 x 25 mm. Based on this, a stick with this dimension contains 4 specimens (25 x 25 x 100 mm), 2 for longitudinal and 2 for transversal absorption. All in all, there were approximately 200 specimens (lesser because of branches and other defects that could distort the result). The samples were brushed with polyurethane glue to isolate the surfaces that should not provide absorption/desorption. In the longitudinal water uptake, every surface was glued except the end- grain surfaces. The specimens for the transversal water uptake were isolated at the end-grain and at two other parallel surfaces. This test continued 20 days. After the absorption experiment, all samples were left in a room climate and the decrease of their mass by desorption was recorded.

## **RESULTS AND DISCUSSION**

### **Determination of the water absorption/desorption of the studied species**

The water absorption of the specimens increased over time. In order to get a linear relationship, the water absorption was defined as a function of the square root of the exposure time. The incline of the water absorption lines is defined as water absorption coefficient  $A_w$ .

As one can see, the values for the Siberian larch from the two origins do not differ significantly. According to the macroscopic investigation (both species with different origins have the same amount of annual rings and quantity of late wood), this result seems legitim. The study also showed that there is no difference for the radial and tangential surface in the transversal water uptake. This is why the results are summarised as transversal adsorption/desorption. Referring to the work of G.Koch (2007), where Siberian larches from various origins showed different susceptibility to decay, it is now evident that also the water absorption coefficient does not play a significant role; the origin does not influence this property. If the density is considered, there is also no clear visible relation: the specimens of the European larch and the pine sapwood show nearly the same density, but there is a significant difference in the absorption behaviour. An interesting thing could be that the relation of the water uptakes between the species differs. The transversal water uptake of the larch species is nearly 30% of the longitudinal, the transversal water uptake of the pine wood in

relation 25%. In this view it could be possible that boards out of pine heartwood are more resistant against rain than larch wood (according to the results for the tangential surface).

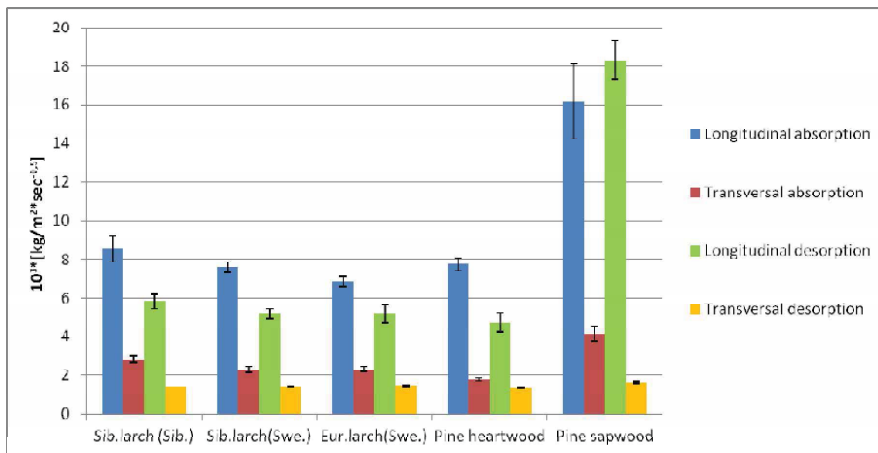


Fig.1: water absorption coefficient of the tested species

### Above ground field test

The upper surfaces of the studied larch species showed no sign of decay after 12 years of exposure above ground. The reason for this could be the faster temperature changing, which leads to a faster (higher) hornification. The bottom surfaces of every species shows some decay, the reason for this could be the higher and more constant moisture content (hanging drops after rain) and deficiency of sunlight. Standing presence of moisture, nearly no light and water contact with the end-grain surfaces determined the lowest decay resistance of the joint area. Having this in mind, the decay rate of only the lap joint area was plotted in the Table 1.

Table 1: Rating of lap-joints according to ENV 12 037 after 12 years above ground exposure

Wood species/years of exposure	1	2	3	4	5	6	7	8	9	10	11	12
Sib. larch (Sib.)	0	0	0	0	0	0	0	0	0	0	0	0
Sib. larch (Swe.)	0	0	0	0	0	0	0	0	0.5	1	1	1
Eur. larch (Swe.)	0	0	0	0	1	1	1	1	2	2	2	2
Pine heartwood	0	0	0	0	0	0.5	1	1	1	1	1	1.5
Pine sapwood	0	0	0.5	1	2	3	4	4				

It is noticeable that the difference between the water uptake of European larch and Siberian larch is not that big, but therefore the decay. A reason for the higher decay resistance of the Siberian larch could be more longitudinal resin channels, which give also more extractives.

*In-ground field test*

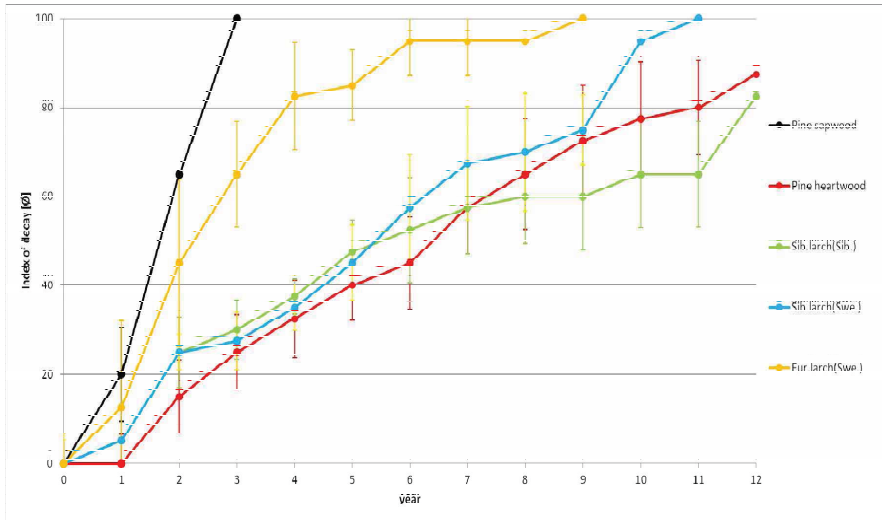


Fig.2.: results from the Ultuna testfield

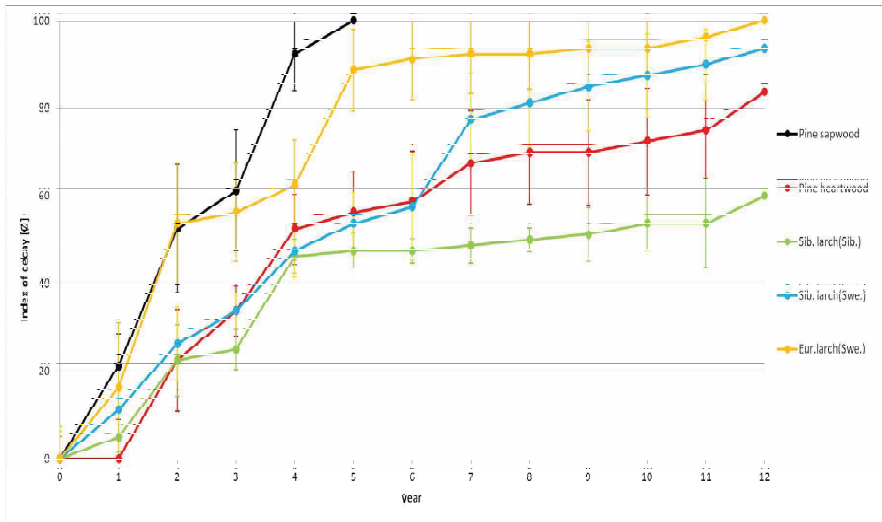


Fig.3: results from the Simlångsdalen testfield

The Siberian larch from the Siberian origin shows in both cases the highest decay resistance. In contrast to the water uptake a clear difference is visible: the Siberian larch from Siberia shows a much higher decay resistance than the larch from Sweden. It is interesting to see the behaviour of the pine heartwood: in both cases the values lay



between the Siberian larch species. With this knowledge one could conclude that the Scots pine heartwood ensures somewhat longer service life than the Siberian and European larch wood from Sweden. For all species one can observe a higher decay in Ultuna. A reason for that could be the higher water holding capacity, the neutral pH value and presence of bacteria as degrader. The decay index in the Figures shows that the European larch is not very durable in ground contact. Referring to the water uptake behavior, we can argue now that the decay resistance stays in no relation with it. The Siberian larch from Siberia and the Siberian larch from Sweden have nearly the same water uptake coefficient, but show a significant difference in the decay rate.

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# EFFECT OF HEAT TREATMENT ON MECHANICAL PROPERTIES AND DECAY RESISTANCE OF WOOD

Navickas, P.<sup>1</sup> & Albrektas, D.<sup>2</sup>

## ABSTRACT

Heat treatment is often used to improve dimensional stability of wood. This research was performed in order to determine how the heating process affects mechanical properties and decay resistance of oak and lime wood. Specimens were subjected to 3 hour heating at 130, 160, 190 and 220 °C temperatures. After undergoing heat treatment, mechanical properties of heated and unheated specimens were tested. The results showed that treated specimens had lower mechanical properties compared to untreated ones. Moreover, the mechanical strength decreased with an increase in the treatment temperature. On the other hand, the higher the heating temperature, the better wood resistance to decay.

Keywords: heat-treated wood, mechanical properties, oak, lime.

## INTRODUCTION

Wood is a complex polymeric material that consists of cellulose, hemicellulose, lignin and extractives. It is one of the strongest and most widely used organic materials. Wood is easily treatable and very significant in the furniture and construction industries. For its properties wood is used in light constructions and in separate cases it can substitute metal elements. Moreover, wood has a good thermal resistance and a good weight and strength ratio. However, regardless of these advantages, wood has a number of drawbacks. Because of humidity and other environmental conditions, the dimensions and humidity of the wood changes, it becomes susceptible to the effects of fungi and biological pests. During the recent decades a number of methods have been tested in order to improve the durability and stability of wood's dimensions avoiding the usage of chemicals. One of these methods is the heat treatment (Aydin et al., 2005; Juodeikienė et al., 2003; Li Shi et al., 2007; ThermoWood 2003).

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The heat treatment is one of wood modification methods with which the dimensional stability of wood is increased and it becomes more resistant to the biological effects. During the heating process chemical changes of lignin and hemicellulose take place within the wood, thus making it less hygroscopic. Temperatures higher than 150 degrees irreversibly alter mechanical and chemical properties of wood. The higher the heating temperature, the better the biological resistance and dimensional stability of wood. Also, the hygroscopicity of wood becomes lower; however, its mechanical properties deteriorate. Wood becomes fragile, its resistance to bending and stretching decreases to 10 – 30 %. During the process of heating the weight of wood also diminishes: the longer it is heated and the higher the temperature, the less it will weigh (Korkut et al., 2009; Syrjanen et al., 2001; Yildiz et al., 2006).

The change of wood's properties during the heating process depends on the heating method, the type of wood and its properties, wood's initial humidity, and the duration and temperature of heating. The heating temperature has a much bigger impact to the properties of wood than the duration of heating. When wood is heated in lower temperatures for a longer period of time uneven results are gained in comparison with the ones when wood is heated in higher temperatures for a shorter period of time (Korkut 2008; Akyildiz et al., 2008).

The aim of this work is to evaluate the heating effect to the mechanical properties and biological resistance of oak and lime wood.

## MATERIAL AND METHODS

Tests involved oak and lime wood specimens with the following dimensions: 300 × 20 × 20mm. Specimens were cut from 1600 × 20 × 20mm planed beams of oak and lime wood. Each beam was used for cutting five specimens and two moisture sections. One group of specimens did not undergo heating and the other group underwent 3-hour heating in air at the following temperatures: 130°C, 160°C, 190°C and 220°C under atmospheric pressure.

After exposure to heat dimensions of specimens were measured (length, width and thickness with accuracy of 0.05 mm, 0.01 mm and 0.01 mm, respectively), specimens were weighed to the nearest 0.01g and their change in mass and volume was established. In order to examine resistance of heated wood to wood-destroying and colouring fungi, one half of specimens were stored in an environment contaminated with the above-mentioned fungi for 10 months at 5 – 15°C temperature and 85-95% relative humidity. Exposure to fungi was recorded by visual observation. The other half of specimens underwent impact bending strength testing and obtained results were converted to 12% moisture basis.

## RESULTS

### Change in Mass and Volume

After exposure to heat measurements and weight of specimens were recorded and their change in mass and volume was calculated. Obtained results reveal that oak specimens tend to be more stable in comparison to lime specimens (Fig.1). When oak specimens underwent heating at 130°C temperature, the volume decreased by 1.7%, whereas, the volume of lime specimens declined by 2% on average at the heating temperature of 130°C, i.e. by almost the same percentage as in the case of oak specimens that were heated at 160°C temperature (1.9%). Only at 220°C temperature a larger decrease in volume of oak wood specimens (4.9%) was observed. In the presence of such temperature the volume of lime wood specimens declined by 5.8%. It was obtained that during the heating process the volume of lime specimens was decreasing faster than the one of oak specimens, however, their mass was declining slower (Fig. 2). An increase in the heating temperature led to a decrease in the mass of specimens. This is mainly related to a decrease in wood moisture and depolymerization reactions of wood polymers during the heating process (Akyildiz 2008). The largest decline in mass was observed in the case of oak specimens that underwent heating at 220°C temperature.

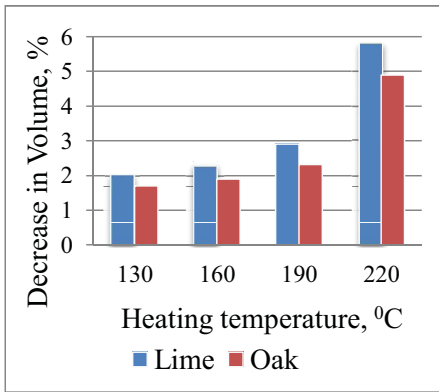


Fig. 1. Heat-induced changes in volume.

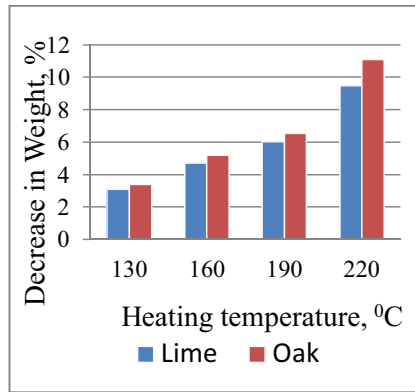


Fig. 2. Heat-induced changes in weight.

After exposure to heat the mass of oak and lime specimens was lower by 11.1% and 9.5%, respectively. At all heating temperatures the mass of oak specimens decreased more than the one of lime specimens, which can be explained by the fact that oak wood has a higher density than lime wood and specimens with lower density show more resistance to thermal decomposition in comparison to specimens with higher density (Korkut et al., 2008). When wood is subjected to thermal treatment, all its components (cellulose, hemicellulose and lignin) are affected. Oak wood contains larger amounts of hemicellulose, which has the fastest rates of decomposition than the other components due to its heterogeneous structure (Chaouch et al., 2010).

## Resistance to Fungal Attack

In the next testing stage specimens were placed in an environment contaminated with wood-colouring and wood-destroying fungi. Specimens were placed into the stand by ensuring their contact with contaminated wood. Exposure to fungi was recorded by visual observation. During the test lime wood specimens were among the most affected by fungi and their effect was first noticed at the ends of specimens. This can be explained by the fact that a relatively large amount of moisture is removed and it enters specimens through their ends (Kajalavačius 1992) by making these areas moister and most suitable for fungal breeding. It was observed that fungi had no significant impact on heated and unheated oak wood specimens during the experiment. Only those oak wood specimens that were unheated and heated at 130°C temperature were affected by mould fungi and wood-colouring fungi (based on visual observations, it can be stated that these fungi belong to the *Cortitium laeve* tribe) (Fig.3), which covered 10% of the surface area of specimens. Lime wood was most affected by fungi, which were steadily and abundantly growing on lime wood specimens that were unheated and heated at 130 – 190°C temperatures. Only those lime wood specimens that underwent heating at the highest temperature, i.e. 220°C, deserve attention, since they showed more resistance to fungal attacks.



Fig. 3. Oak wood specimens affected by fungi.



Fig. 4. Lime wood specimens affected by fungi.

The analysis of specimens revealed the effect of pink-, yellow- and blue-stain fungi as well as wood-destroying fungi (Fig. 4). The ends of specimens were most affected by wood-destroying fungi, which were steadily and abundantly growing on lime wood specimens that were unheated and heated at all temperatures. A lower degree of fungal development was observed only in the case of lime wood specimens that were exposed to 190 – 220°C temperatures.

Wood-destroying fungi covered approximately 10-20% of the surface area of specimens, whereas, wood-colouring fungi affected about 50 – 80% of the surface area of lime wood specimens that were unheated and heated at 130 – 190°C temperature, and 10 % of the surface area of specimens that were heated at 220°C temperature.

### Mechanical Resistance

In order to determine the heating effect to the mechanical properties of wood the testing of impact bending strength and impact hardness was performed. The results were recalculated into a 12% humidity of specimens. The gained results show that the higher the heating temperature, the lower is the resilience of specimens (Fig. 5). Also, while increasing the heating temperature this parameter was decreasing faster for oak specimens than for the lime specimens. The resilience of lime specimens heated in the temperature of 130°C decreased by almost 6%, while the resilience of oak specimens heated in the same temperature dropped by 16%. The resilience of oak specimens heated in 220°C was 54% while the resilience of lime specimens was 45% lower in comparison with the unheated specimens. After heating in the temperature of 220°C the resilience of oak specimens was 6% higher if compared to the lime ones, while for the unheated specimens it was 22% higher. The decrease of wood's strength properties is related with the degree of thermal fragmentation and the loss of weight after heating, but mainly it depends on the depolymerization of wood's polymers.

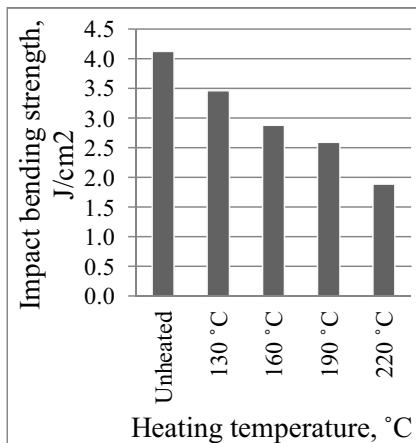


Fig. 5a. Impact bending strength of oak

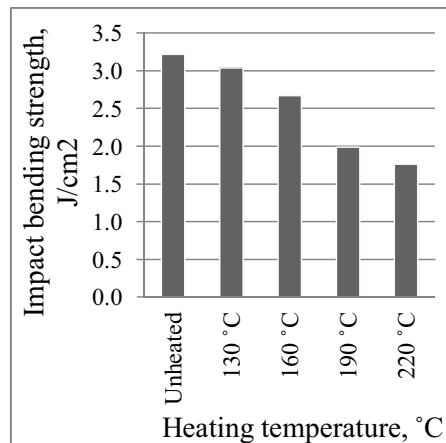


Fig. 5b. Impact bending strength of lime

The main reason why wood loses its strength is the decomposition of hemicellulose which is less stable and more susceptible to the higher temperatures than cellulose or lignin. Changes in hemicellulose or its total dissociation are the most significant to the wood's strength after the heating process (Korkut 2008). Since there is more hemicellulose in oak than in lime, during the heating process the strength properties of oak specimens were decreasing faster.

## CONCLUSIONS

1. It was obtained that a decrease in volume, density and mass of heated specimens is proportional to the heating temperature: an increase in temperature leads to significant changes.
2. It was established that after exposure to 220°C temperature the largest decrease in mass was observed in the case of oak specimens. The difference between decreased masses of oak and lime wood specimens that were heated at this temperature was 14.6%.
3. The largest growth of wood-colouring fungi and mould fungi was observed at the ends of lime wood specimens that were unheated and heated at 130 – 160°C temperature.
4. An increase in the heating temperature led to a decrease in impact strength of specimens. The largest decline was recorded, when oak and lime specimens underwent heating at 220 °C temperature (27%) and 160°C temperature (25%), respectively.
5. Wood heating can be used, when it is necessary to reduce wood hygroscopicity and to enhance its biological resistance.

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# INFLUENCE OF THERMAL TREATMENT ON THE STATIC BENDING STRENGTH OF BLACK ALDER WOOD

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## ABSTRACT

Effect of thermal treatment on the bending strength of black alder wood was examined. The wood samples were treated at temperature of 60, 80 100 and 120°C and duration for 12, 24, 48 and 96 hours. It was determined that bending strength was decreased as the treatment temperature and duration was increased. Effect of heating temperature and duration on static bending strength depends on temperature. At lower (60–80°C) temperatures, influence of heating duration was more intensive, but at higher temperatures (100–120°C) more intensive effect of temperature was found. After thermal treatment average decrease of bending strength was found approximately 8 %. After heat treatment moisture of black alder wood decreases twice, while thermal treatment at relatively low temperatures does not influence on density changes.

Key words: black alder wood, thermal treatment, bending strength, density, moisture content.

## INTRODUCTION

Wood is one of most widely used materials. It is natural, ecological and can be used for various purposes. It can be used as constructional material in building industry, for interior and exterior elements, furniture, transport, packaging industry, etc. Great part of wood is used for artificial fibres and paper production (Morkevičius and Papreckis 2004). The decrease of total forests area, results on the decrease of high quality wood recourses and nowadays more and more important become investigations based on the improvement lower quality wood properties. One of biggest disadvantages of wood is sorption environmental humidity and its evaporation. The variation of wood moisture content results on the measurement changes. Besides, damp wood usually mould and

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decay. In order to eliminate these problems various modes of wood modification are used.

One of wood properties improvement modes is thermal treatment or heating. Compare to wood chemical impregnation, this technology is good and environmentally friendly (Korkut, Akgul and Dundar 2008). Thermal modification can significantly increase wood biological durability, resistance to biological pests and improve measurements stability (Juodeikienė and Minelga 2003; Tornainen, Dagbro and Moren 2011). On the other hand, after this modification some properties can decrease (Juodeikienė 2009). For example, wood fragility increases, bending and tension strength decreases in the range of 10-30 % (Ates, Akyildiz and Ozdemir 2009; Korkut, Kok, Korkut, and Gurleyen 2008). It was pointed, that beginning of wood structural changes appears during heating at lower temperatures, which are close to drying temperatures (Heräjärvi, H. 2009; Källander and Bengtsson 2003; Sehlstedt-Persson 2005). That is due to changes of chemical structure of wood (Ates, Akyildiz and Ozdemir 2009).

In the last decade the increase of black alder wood application in building industry (especially for sauna building), furniture production is observed. That is because this kind of wood can be easily processed, does not have high deformability during drying, and is resistant in water and humid environment. Besides, black alder is one of most popular leafy wood kinds in Lithuania. It covers about 6.5% of total forest area.

Independently on the high industrial importance of this kind of wood the research based on the investigation properties of black alder wood are quite limited up to now. Due to that aim of this investigation was to find out influence of thermal treatment on the black alder wood static bending strength, humidity and density.

## MATERIAL AND METHODS

For investigations black alder (*Alnus glutinosa* L.) wood from Jurbarkas region, Lithuania was used. Blanks with measurements of 35×45×1500 mm and initial moisture content of 23 %, were putted together in to small piles and stored inside up to moisture decreased down to 10–11 %. After that samples for tests were cutted. Crossection of samples was 20×20 mm and length was 300 mm.

Samples were heated at temperature of 60, 80, 100 and 120°C for 12, 24, 48 and 96 hours under atmospheric pressure. The number of samples for mechanical tests was determined according to the requirements of ISO 3129–1975. To obtain result of bending test, for each point group from 36 samples was tested. Amount of annual rings per centimetre of sample crossection was calculated using optical microscope MIKKO. The density and moisture of samples were determined according to the requirements of standards ISO 3131–1975 and ISO 3130–1975 respectively. Before mechanical tests samples (treated and untreated) were conditioned for 4 weeks at a temperature of 20±2 °C and the relative humidity of the atmosphere 65±2 %.

Static bending test was performed according to the requirements of standard ISO 3133–1975 using testing machine R-0.5 working at static mode. Test was carried out slowly increasing bending force. Determined strength was recalculated to static bending strength of wood with moisture of 12%. Only recalculated results are discussed in this paper.

## RESULTS AND DISCUSSION

The moisture and density of untreated and thermally treated black alder wood samples are presented in Table 1 and Table 2.

**Table 1.** Moisture (%) of untreated and thermally treated black alder wood samples

Treatment duration	Temperature, °C				
	Untreated	60°C	80°C	100°C	120°C
Untreated	10				
12 h		6	5	4	4
24 h		6	5	4	4
48 h		5	5	4	4
96 h		5	4	4	4

The humidity of thermally treated samples was 54.4% lower compare to those of untreated.

**Table 2.** Density (kg/m<sup>3</sup>) of untreated and thermally treated black alder wood samples

Treatment duration	Temperature				
	Untreated	60°C	80°C	100°C	120°C
Untreated	521				
12 h		523	504	497	500
24 h		508	514	504	494
48 h		493	511	505	500
96 h		506	486	491	498

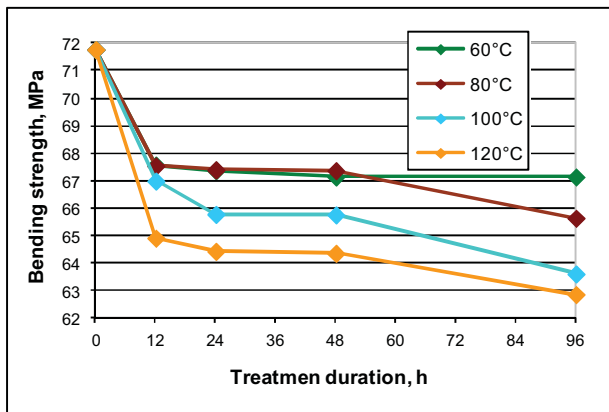
Density of thermally treated samples was about 3,2% (average density was 502 kg/m<sup>3</sup>) lower compare to those of untreated samples (521 kg/m<sup>3</sup>). Otherwise, it was not determined density changes during variation of temperature or thermal treatment duration. That allows to the conclusion that thermal treatment at lower temperatures does not influence on the density changes of black alder wood. While other authors indicate that at high temperatures (above 200°C) decrease of wood density can be found. For example, after heat treatment of Calabrian pine (*Pinus brutia* Ten.) wood at 230°C temperature for 2-8 hours density decreased 10-15 % (Ates, Akyildiz and Ozdemir 2009).

Independently on the insignificant density changes of black alder wood after heating at lower temperatures, the changes in colour, similar to those obtained at high temperatures was found (Ates, Akyildiz and Ozdemir 2009). The higher heating temperature and duration the darker colour of samples occur (Fig. 1.).

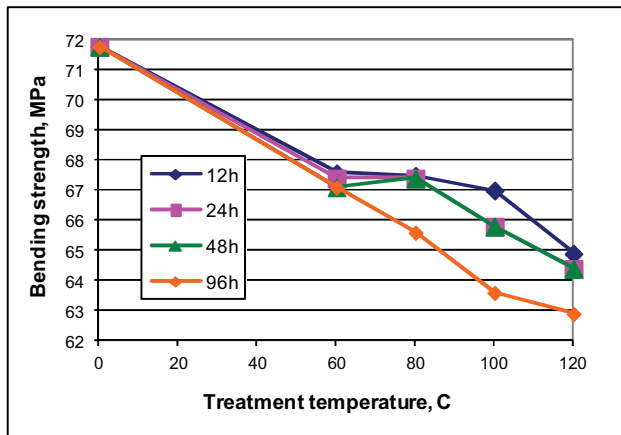


**Fig. 1.** Colour changes vs increase thermal treatment temperature and duration

The influence of thermal treatment temperature and duration on the black alder wood static bending strength is presented in Fig. 2 and Fig. 3.



**Fig. 2.** Influence of heat treatment duration on the static bending strength of black alder wood.

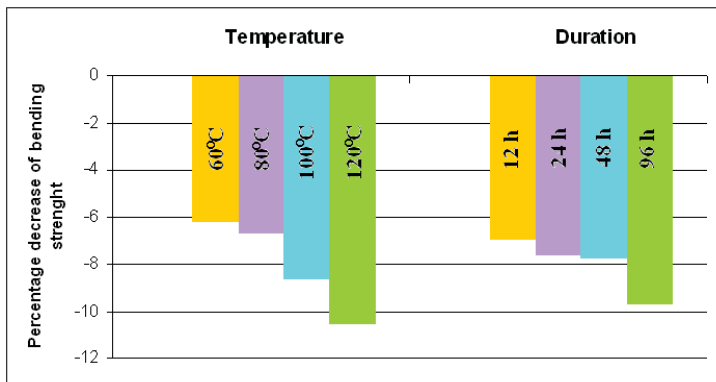


**Fig. 3.** Influence of heat treatment temperature on the static bending strength of black alder wood.

During evaluation influence on the thermal treatment duration it was found that after 12 hours of treatment average bending strength decreased 7 % (66.8 MPa), after 24 hours it decreased 7.7 % (66.3 MPa), after 48 hours – 7.8 % (66.2 MPa), and after 96 hours of thermal treatment it decreased 9.7 % (64,8 MPa) compare to the strength of untreated samples (71.8 MPa).

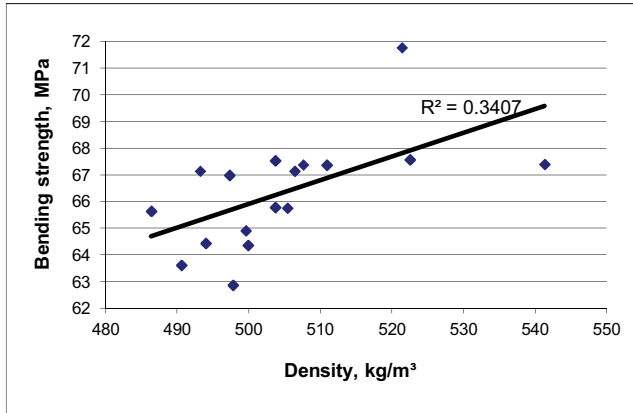
Influence of thermal treatment on the static bending strength of black alder wood is as follows: after heating at temperature of 60°C samples bending strength decreased 6.2 % (67,3 MPa), at 80°C – 6.7 % (67 MPa), at 100°C – 8.7 % and after heating at temperature of 120°C bending strength decreased 10.6 % (64.2 MPa). Obtained results confirmed results of other authors obtained with other kinds of wood. Static bending strength of pine wood (*Pinus sylvestris* L.) after treatment at temperature of 120°C for 10 hours decreased 9.6 % (Korkut, Akgul and Dundar 2008), of hazelnut wood (*Corylus colurna* L.) that was 6 % (Korkut and Hiziroglu 2009). Static bending strength of calabrian pine (*Pinus brutia* Ten.) wood after treatment at temperature of 130°C for 2 hours decreased 18 % (Ates, Akyildiz and Ozdemir 2009).

As it can be seen, both thermal treatment temperature and duration has similar influence on the bending strength: in both cases the bending strength decreased approximately about 8%. On the other hand, during separate evaluation of temperature and duration effect is evident that at lower temperatures (60–80°C), more significant effect of treatment duration is expressed, and at temperatures from 100°C up to 120°C bending strength changes is more influenced by treatment temperature. The higher heating temperature and heating duration the lower bending strength was found (Fig. 4.).



**Fig. 4.** Percentage expression of changes in bending strength as a function of treatment temperature and duration.

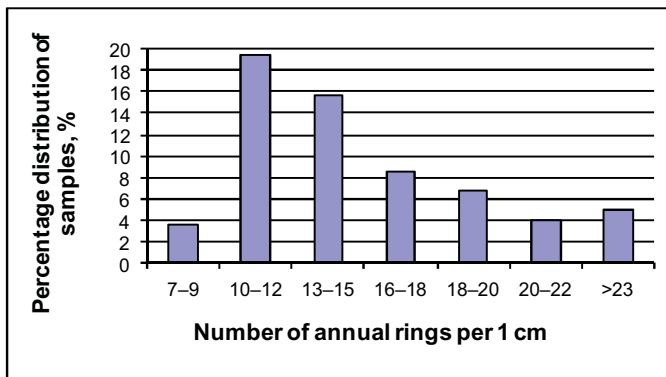
During investigation was also determined that density also influences on the bending strength of samples. The higher samples density the higher bending strength is observed (Fig. 5.).



**Fig. 5.** Black alder wood static bending strength vs density.

It is believable, that mechanical properties of wood also are dependent on the width of annual rings, or particularly on the part of the latest wood in the ring, i.e. on the part which highly influences density of the wood. As the annual rings of black alder wood are unclear, it is very difficult to determine amount of latest wood in the ring. Due to that it was calculated number of annual rings in the one centimetre of cross-section. In Figure 6 is presented percentage distribution of tested samples in respect to number of annual rings. Evident, that 50 % of all samples consists from 10 up to 20 annual rings per cm.

In Figure 7 is shown relation between number of annual rings per 1 cm of cross-section and static bending strength.



**Fig. 6.** Percentage distribution of samples used for static bending test vs amount of annual rings.

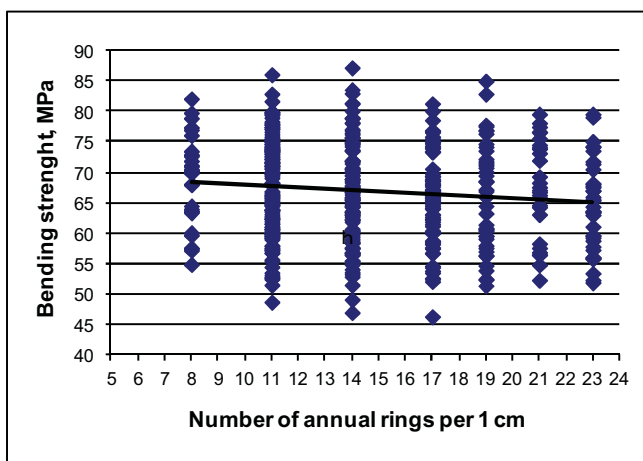


Fig. 7. Influence of number of annual rings on the bending strength of black alder wood

For black alder wood as for other kind of wood is characteristic optimal number of annual rings at which best mechanical properties are reached. In all other cases, i.e. at narrow or at wide rings, this indicates increase or decrease number of annual rings, mechanical properties starts to decrease. In our investigated case highest static bending strength values for black alder wood was found when number of annual rings per centimetre was 9. Stress tends to significant decrease when number of annual rings per centimetre is 15. In the literature also can be found data indicating optimal rings number and width of annual rings for other kinds of wood. For high quality oak wood number of annual rings in 1 cm must be not higher than 12, for ash that is 12 (Jakimavičius 2008).

## CONCLUSIONS

1. After heat treatment at temperature from 60 up to 120°C moisture of black alder wood decreases up to 54.4 %.
2. Influence of thermal treatment at temperature from 60 up to 120°C on the density changes of black alder wood is insignificant.
3. After thermal treatment average decrease of black alder wood bending strength is about 8%. After thermal treatment at temperatures from 60°C up to 80°C more significant effect of treatment duration was found. At the increase of heating temperature more significant effect of temperature was expressed.
4. The bending strength of black alder wood is dependent on the density: the increase of density results on the increase of bending strength (correlation coefficient was  $r=0.58$ ).
5. The highest bending strength was found at number of annual rings of 9. When number of annual rings is bigger than 15 bending strength drastically decreases.

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# PREDICTION OF BENDING PROPERTIES OF IMPREGNATED SMALL DIAMETER SCOTS PINE POSTS USING ACOUSTIC VELOCITY

Möttönen, V.<sup>1</sup>, Heräjärvi, H.<sup>2</sup>, Stöd, R.<sup>3</sup> & Koivunen, H.<sup>4</sup>

## ABSTRACT

The aim of the study was to compare the modulus of elasticity (MOE) and modulus of rupture (MOR) of untreated and preservative impregnated small diameter Scots pine (*Pinus sylvestris*) fencing posts in static bending test. Butt logs originating from young and advanced thinning stands were turned to the nominal diameter of 120 mm and cross cut to the length of 2350 mm. The impregnation treatments were pressure impregnation to AB class and pine oil impregnation. After the relatively mild four-week-long weather chamber test, the posts were stored in room temperature until acoustic and destructive bending tests. The acoustic velocity was measured with Director HM200 tool. Dynamic MOE of the test posts was calculated based on the acoustic velocity and wood density, and compared with the bending MOE and MOR values obtained from the static bending tests (EN 408:2003). In calculations, the MC's of the posts were adjusted to correspond to 12%. The untreated posts had the highest acoustic velocity, 4.9 km/s. The acoustic velocity did not differ between the pressure impregnated and pine oil impregnated posts, being 4.6 km/s in both groups. Linear regression analysis showed a significant relationship between the static and dynamic MOE for the entire data ( $r^2$ : 0.78), the  $r^2$  values for different treatment groups varying from 0.40 to 0.91. Linear regression analysis between the MOR and dynamic MOE resulted in  $r^2$  values from 0.01 to 0.74. Regarding the dependence on wood density, the  $r^2$  values of 0.00-0.87 and 0.07-0.74 were obtained for the static MOE and MOR, respectively. In all cases, the untreated posts showed higher  $r^2$  values than pressure impregnated or pine oil impregnated posts. To conclude, chemical modification of wood appears to decrease its acoustic velocity. However, the acoustic velocity seems to

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be an applicable method to predict the bending MOE of both untreated and chemically modified posts.

Key words: Acoustic measurement, Pine oil treatment, Pressure impregnation, Stiffness, Strength

## INTRODUCTION

The volume, annual growth and annual harvesting removal of Scots pine (*Pinus sylvestris* L.) timber in Finland are 1098, 45, and 23 million m<sup>3</sup>, respectively (Ylitalo 2011). Sequential thinnings of forest stands at least twice during the rotation period belong to the modern forestry practice in Finland. At the time of the first thinning, the log diameter and quality do not fulfil the requirements of logs for traditional saw mill production as far as structural products and carpentry are concerned. A growing proportion of harvested timber originates from the first or second commercial thinning stands, which means that the average volume of logs gets smaller than is the case in conventional final felling stands. Utilisation of small diameter logs has been increased, e.g., in small scale and garden construction (e.g., Boren 1999, 2001; Ranta-Maunus 1999, Wall *et al.* 2005). However, there are industrial sawmills using logs as small as 70 mm in top diameter. Such raw material can be processed economically only if the production efficiency is very high; for instance, the saw line speed is typically 150-180 m/min, logs are run almost without log-to-log spaces (fixed sawing pattern), and the lines are virtually unmanned. The current products made of small diameter logs include e.g., moldings and lamellae for glulam boards. However, there is a global and growing need to find new economically viable and high quality wood products that can be manufactured from small sized log material, both from softwood and hardwood species (see: Spelter *et al.* 1996, Boren 1999, Kilpeläinen *et al.* 2011).

Small log volume means not only demands of increased efficiency for material handling in logistics and manufacturing processes, but also challenging wood properties such as high proportion of juvenile wood, knotty wood, and sapwood. Due to these facts, products made of small diameter logs are prone to twist and check, and have poor durability against weather. In addition, stiffness and strength of products made of small diameter timber is supposed to be lower than those of products originating from larger logs (e.g., Bodig & Jayne 1982, Zobel & van Buijtenen 1989, Ranta-Maunus 1999, Boren & Barnard 2000, Heräjärvi *et al.* 2000, Boren 2001, Wall *et al.* 2005, Stöd & Kilpeläinen 2006, Stöd *et al.* 2006).

The need for an increase in the utilisation of small diameter logs in structural uses means higher demand for controlling and modifying the challenging wood properties such as high proportion of juvenile wood and sapwood. In addition to the physical challenges, there are chemical ones, as well. Products with increased water resistance and resistance against decay fungi and mould growth are needed. The most common method to preserve wood against microbial growth is pressure impregnation with chromium-copper (CC) based additives. Finland alone produces more than 200,000 m<sup>3</sup>

of CC-impregnated sawn timber annually (Production statistics...2012). In comparison to the copper-chromium-arsene based preservatives that were widely used still in the 1990's, the current CC-preservatives contain less chemicals harmful to the environment.

Pine oil (or "crude tall oil") impregnation, either alone or combined with some other modification such as heat treatment, has been considered as an alternative for CC-impregnation (e.g., Cartwright & Findley 1958, Banks 1973, Sailer *et al.* 1998, 2000, Paajanen *et al.* 1999, Van Eckeveld 2001, Van Eckeveld *et al.* 2001, Paajanen & Ritschkoff 2002, Koski 2008). According to the experience of industry representatives, pine oil penetrates in wood as its best via sawn or peeled surfaces, whereas planed surfaces are more challenging to impregnate. The time period between surface machining and impregnation also appears to have an influence on the impregnability, which is in line with the experiences from the glue joint behaviour.

The dynamic and static modulus of elasticity (MOE) are known to correlate with each other (e.g., Smulski 1991, Ilic 2001, Divos & Tanaka 2005, Liang & Fu 2007). Therefore, the sawmilling industries have utilised acoustic tools for lumber assessment for decades. In addition to structural lumber, acoustic nondestructive testing (NDT) devices have been applied for the evaluation of mechanical properties of poles, pulpwood, veneers, as well as decay detection of growing trees, tree selection and breeding based on stiffness (e.g., Matheson *et al.* 2002, Huang *et al.* 2003, Dickson *et al.* 2004, Edlund *et al.* 2006, Grabianowski *et al.* 2006, Wang *et al.* 2007a,b, Amishev & Murphy 2008, Anttonen 2010, Widmann & Beikircher 2010). Recently, acoustic measurement tools have been tried to implement in harvester heads in order to assess wood stiffness real-time during the logging operations (e.g., Carter 2007, Amishev & Murphy 2008, Amishev *et al.* 2010). Despite of the various studies made for green or dried solid wood, not much is known about the sound velocity in impregnated wood. Acoustic measurements could be a solution for strength grading of impregnated wood products, as well.

The objective of this study was to investigate the acoustic velocity as a tool to predict bending MOE and MOR of non-treated, pressure impregnated and pine oil impregnated round small diameter Scots pine (*Pinus sylvestris*) fencing posts.

## MATERIALS AND METHODS

The posts were divided into following treatment groups:

- 1) Pine oil type 1A impregnation using the process of Ekopine Ltd, posts peeled and impregnated after air-drying of logs for nine months (7 posts),
- 2) Pine oil type 1B impregnation using the process of Ekopine Ltd, posts peeled and impregnated in green state (5 posts),
- 3) Pine oil type 2 impregnation, posts peeled and impregnated after air-drying of logs for nine months (6 posts),

- 4) Commercial copper-chromium based pressure impregnation to AB class for green posts (6 posts),
- 5) Untreated 1, posts peeled after air-drying of logs for nine months (6 posts),
- 6) Untreated 2, posts peeled in green state (5 posts).

The study material for treatment groups 1,3,4 and 5 originated from five thinning stands of Scots pine (*Pinus sylvestris*) in south-eastern Finland, which were harvested in the beginning of year 2009. All logs included in these materials were butt logs with a top diameter less than 155 mm over bark. The reference materials for treatment groups 2 and 6 were of unknown detailed origin, only information being that they are Scots pine from a thinning stand in North-Eastern Finland. Altogether 35 round posts with nominal diameter of 120 mm and length of 2280 mm were peeled from the logs.

The difference between pine oils type A and B was in composition and amount of thinner, which was used to get differences in the viscosity and absorption of pine oil. Pine oil A impregnated posts had been dried slightly before the impregnation, but the moisture content was still above the fibre saturation point (Table 1).

After the impregnation, mass, dimensions, deformations, checks, as well as other possible defects were measured from the specimens. Then, they were subjected to a relatively mild weather exposure test in a weather chamber (Table 2). By this, we wanted to create slightly more realistic results concerning, *e.g.*, checking and warping of posts, that probably have an influence on the acoustic velocity readings as well as the static bending performance. The cyclic weather chamber test lasted 14 days, and consisted of seven successive cycles with two phases. In the conditions of Phase 1, the EMC of untreated wood should settle down to approximately 21%. The conditions in Phase 2 should get the EMC of untreated wood down to 4.2%. Since the weather chamber tests were relatively mild, we assume that the wanted EMC values were not likely achieved. As a result, the checking and warping behaviour of the specimens was quite moderate.

After the weather chamber test, the posts were stored in room temperature until the acoustic and destructive bending tests took place.

**Table 1.** Numbers of specimens and their average moisture contents in different treatment groups.

Treatment nr	N of test specimens	Impregnation chemical	MC of wood at the time of treatment [%]
1	7	Pine oil A	35.2
2	5	Pine oil A	42.9
3	6	Pine oil B	41.8
4	6	Cu/Cr (AB class)	41.7
5	6	Untreated	38.0
6	5	Untreated	42.2

**Table 2.** Temperature and humidity conditions during the weather chamber test cycles. The weather chamber tests consisted of seven successive cycles, the total time being two weeks.

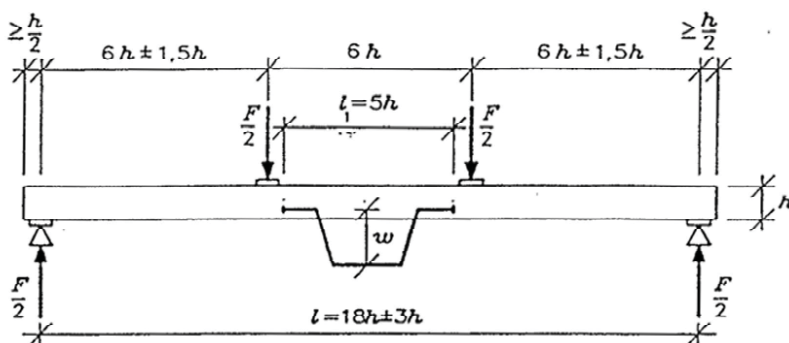
Time [h]	T [°C]	RH [%]
0–36	10	95
37–48	40	15

Air-dry density of wood at the time of acoustic measurement was calculated based on measurements of mass and MC of the posts after the pressure impregnation and pine oil treatments, as well as at the time of the acoustic velocity measurements. The MC of wood was measured using a resistance moisture meter (GANN HT85T). The acoustic velocity was defined as a mean value of two consecutive measurements made using Director HM200 tool. The dynamic MOE of the test posts was calculated according to equation 1:

$$E_{dyn} = v^2 \rho_{12} \quad (1)$$

where  $E_{dyn}$  = dynamic MOE [GPa],  $v$  = acoustic velocity [m/s], and  $\rho_{12}$  = air-dry density [ $\text{kg/m}^3$ ] of wood.

The dimensions, as well as the moisture content (MC), were measured from the posts prior to the bending tests using a calliper and a resistance moisture meter, respectively. In addition, the size and location of knots, checks and other defects were recorded. The rotational position of the posts in bending test was decided based on its defects. The weakest side was visually determined, and marked in the post using a felt-tipped pen. The strength related defects were measured or evaluated from the mid section of the post at 1/3 length. Thus, we concentrated in the section with maximum bending momentum during the four-point bending test (see: Fig. 1). The weakest side was positioned downwards in the bending test. Therefore, the destructive bending results indicate the expected lowermost strength value for each individual post.



Source: Finnish Standard Association SFS

**Fig 1.** Experimental setup in static four-point bending test according to SFS-EN 408 (2003).

Static bending tests were made according to standard EN 408 (2003) using TIRAtest 28100 material testing device of Savonia University of Applied Sciences, Kuopio, Finland. Static MOE was calculated according to equation 2:

$$E_{m,g} = \frac{l^3(F_2 - F_1)}{bh^3(w_2 - w_1)} \left[ \left( \frac{3a}{4l} \right) - \left( \frac{a}{l} \right)^3 \right] \quad (2)$$

where  $E_{m,g}$  = static MOE (global) [GPa],  $l$  = distance between the lower supports [mm],  $b$  = specimen width [mm],  $h$  = specimen height [mm],  $a$  = distance between the load point and the closest support point [mm],  $F_2 = 0.4F_{max}$  [N],  $F_1 = 0.1F_{max}$ ,  $w_2$  = displacement at the point  $F_2$  [mm],  $w_1$  = displacement at the point  $F_1$  [mm].

Static MOR was calculated according to equation 3:

$$f_m = \frac{aF_{max}}{2W} \quad (3)$$

where  $f_m$  = static MOR at 12 % MC [MPa],  $a$  = distance between the load point and the closest support point [mm],  $F_{max}$  = maximum load [N],  $W$  = section modulus, [mm<sup>3</sup>]. The section modulus for round shaped specimens was calculated according to equation 4:

$$W = (\pi r^3)/4 \quad (4)$$

where  $W$  = section modulus,  $\pi$  = the pi factor, and  $r$  = radius of the round post.

For the calculation of the static MOE and MOR, the MC's of the test posts were adjusted to correspond with 12% according to equations 5 and 6 (Boström 1994):

$$E_{m,g,12} = \frac{E_\omega}{1+0.0143(12-\omega)} \quad (5)$$

$$f_{m,12} = \frac{f_\omega}{1+0.0295(12-\omega)} \quad (6)$$

where  $E_{12}$  = static global MOE at 12% MC [GPa],  $\omega$  = MC at the time of the bending test [%],  $E_\omega$  = static global MOE at the MC of  $\omega$  [GPa],  $f_{m,12}$  = static MOR at 12% MC [MPa],  $f_\omega$  = static MOR at the MC of  $\omega$  [MPa].

In this paper, the results are analysed using Pearson correlation analysis and linear regression.

## RESULTS AND DISCUSSION

Untreated posts had the highest acoustic velocity, and the velocities did not differ between pressure impregnated and pine oil impregnated specimens. An exception was made by the pine oil 1 impregnated posts peeled and impregnated in green state, which had the highest acoustic velocity of all treatments.

At the time of the bending test, the MC of wood in pressure impregnated specimens was clearly higher than in pine oil treated and untreated specimens. However, the MC was measured using a resistance meter, thus being probably relatively inaccurate and not fully comparable between the treatments. The differences in the absorption of the impregnation chemical was reflected to the differences in wood density. Pine oil treated specimens had the highest wood density. Densities did not differ between the pressure impregnated and untreated posts. Hence, the pine oil impregnation increased markedly the mass of posts whereas the pressure impregnation did not have any apparent effect on wood density. The dynamic MOE of pressure impregnated posts was lower than that of pine oil treated and untreated posts. Impregnation treatments decreased the capability to predict the static MOR and MOE using the dynamic MOE. However, the correlation between dynamic MOE and static MOE was relatively clear in all treatments (Fig. 2). Widmann & Beikircher (2010) found a strong linear relationship ( $r^2$ : 0.88) between the dynamic and static MOE of thermally modified beechwood boards and beams. However, the dynamic MOE explained only 22% of the variation in MOR in their material.

The Pearson correlation coefficients (Table 3) indicated a strong dependence between dynamic and static MOE in all treatment groups except the CC-impregnated specimens.

The high temperature associated with low MC during the pine oil impregnation process probably causes some permanent damages in the structure of wood tissues. This can explain the good MOE values but relatively low MOR values in pine oil treated specimens. The same phenomenon is familiar from thermally modified timber (*e.g.*, ThermoWood® Handbook 2003, Heräjärvi 2009).

The posts that were peeled in green state appeared to have higher strength than the ones peeled after nine-month drying period. However, the difference may as well be caused by some inherent quality differences between the raw materials. This is reasoned by the fact that the average air-dry density was slightly higher in untreated specimens that were peeled in green condition compared with the untreated specimens stored for nine months before peeling (492 vs. 468 kg/m<sup>3</sup>, respectively). On the other hand, it is possible that the volumetric pine oil absorption was greater in specimens that were peeled after nine-month drying period, resulting in higher relative density increment than in the other specimens. Nevertheless, there appears to be a negative correlation between the sound velocity and density increment caused by pine oil impregnation.

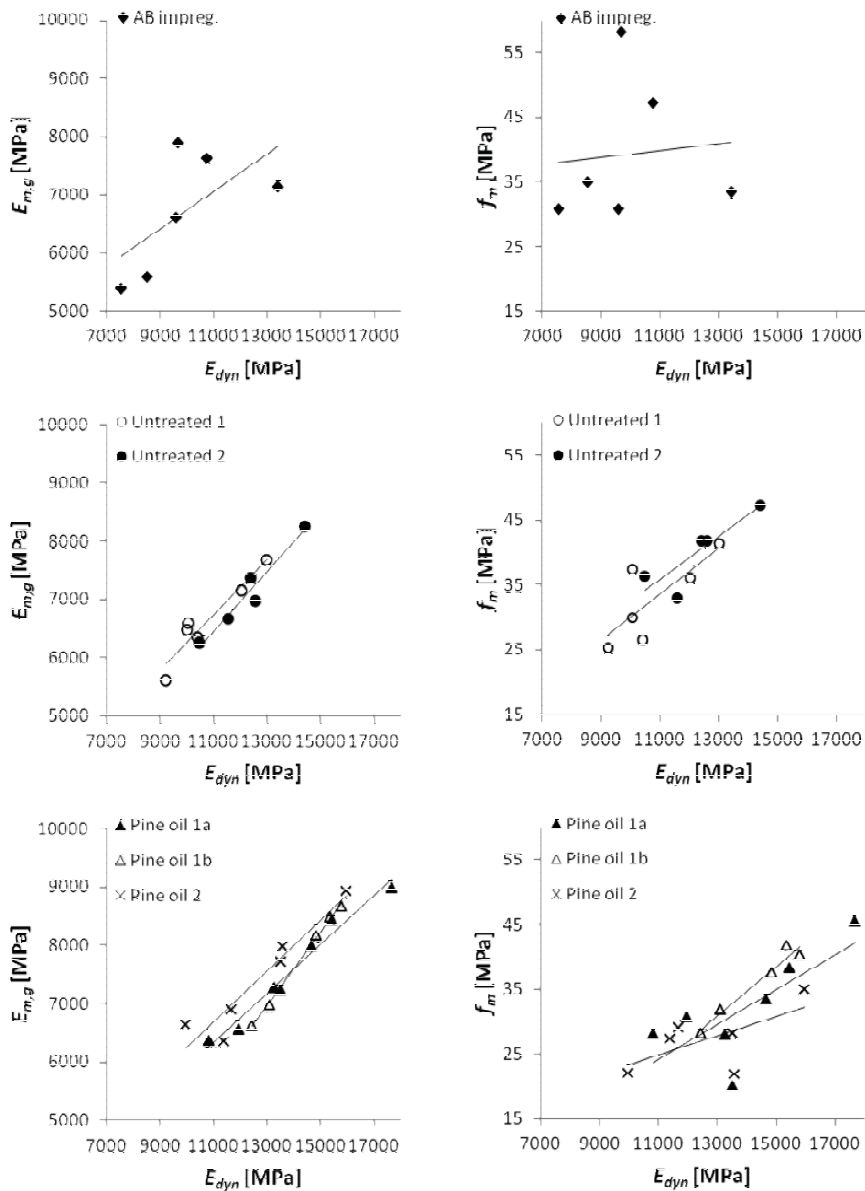
**Table 3.** Pearson correlation coefficients between static MOR, MOE, dynamic MOE, acoustic velocity and air dry density of posts by treatment. \* stands for  $01 < p < 0.05$ , \*\* stands for  $p < 0.01$ .

	$f_{m,12}$	$E_{m,g,12}$	$E_{dyn}$	$\rho_{12}$
Pine oil impregnation 1 – pre-dried (N=7)				
$E_{m,g,12}$	0.769*			
$E_{dyn}$	0.749	0.987**		
$\rho_{12}$	0.524	0.556	0.572	
$v$	0.657	0.945**	0.954**	0.306
Pine oil impregnation 1 – fresh-peeled (N=5)				
$E_{m,g,12}$	0.982**			
$E_{dyn}$	0.979**	0.999**		
$\rho_{12}$	0.798	0.777	0.754	
$v$	0.817	0.857	0.875	0.343
Pine oil impregnation 2 (N=6)				
$E_{m,g,12}$	0.505			
$E_{dyn}$	0.646	0.950**		
$\rho_{12}$	0.245	0.357	0.421	
$v$	0.545	0.786	0.841*	-0.180
CC-impregnation (N=6)				
$E_{m,g,12}$	0.761			
$E_{dyn}$	0.095	0.631		
$\rho_{12}$	0.353	0.715	0.832**	
$v$	-0.116	0.438	0.889*	0.488
Untreated – pre-dried (N=6)				
$E_{m,g,12}$	0.851*			
$E_{dyn}$	0.777	0.955**		
$\rho_{12}$	0.849*	0.946**	0.869*	
$v$	0.519	0.741	0.890*	0.549
Untreated – fresh-peeled (N=5)				
$E_{m,g,12}$	0.872			
$E_{dyn}$	0.866	0.971**		
$\rho_{12}$	0.883*	0.905*	0.833	
$v$	0.465	0.636	0.774	0.298
Entire material (N=35)				
$E_{m,g,12}$	0.475**			
$E_{dyn}$	0.216	0.881**		
$\rho_{12}$	-0.272	0.450**	0.515**	
$v$	0.461**	0.394*	0.460**	-0.518**

In the calculation of dynamic MOE (see: Equation 1), the wood density has been computationally fixed to correspond to average MC of 12%. The sound velocity, on the other hand, was measured just before the destructive bending tests when the MC of wood varied from 6.6 to 31.7 per cent. Moisture content has an influence on sound velocity (e.g., Steiger 1995, Unterwieser & Schickhofer 2011). Here, we did not apply the moisture corrections presented by Steiger (1995). Thus, we assumed that the



differences in wood MC did not have any apparent effect on sound velocity readings, and subsequently, dynamic MOE results.



**Fig 2.** Dependence of static MOE (left side graphs) and MOR (right side graphs) on dynamic MOE and the linear regression lines by treatment.

## CONCLUSIONS

Based on the results of this study, we can state that the static MOE can be predicted for non-treated and pine oil impregnated round fencing posts using acoustic velocity. The prediction models will be presented in our future works.

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# TESTING TENSILE AND BENDING STRENGTH ON TWISTED AND FLUTED BIRCH DOWELS

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## ABSTRACT

The purpose of this research was to find out which kind of dowel works better under tensile and bending strength. In this case, it is important to ensure combining strength and durability of various components forces. The dowel must be the exact diameter and the hole drilled in the best possible fit. To find out these tests results, the author had to work out how he could test the tensile and bending strength by eliminating outside factors. First, details from birch were made, then glued together using two different kind of dowels, sizes 8 x 40 mm and 10 x 40 mm. The test results of tensile strength showed that fluted birch dowel is to some extent better than a twisted dowel. The reason that smooth dowel is stronger is because the grooves are deeper and the glue between detail's surface and dowel's surface helps to bind their grains better. The test results of bending strength showed assuredly that a twisted dowel is stronger than a fluted dowel. This is caused by how they are made. In the process of making twisted dowels wood becomes denser as it is rolled through 3 rollers which create the grooves. The technology to make fluted dowels is much simpler. A milling machine just cuts the grooves into a stick. No high pressure is being used.

**Key words:** wooden dowels, birch, tensile and bending strength analysis

## INTRODUCTION

A wooden dowel is called a cylindrical stick, which is used to connect various pieces of wood. The use of dowels has been used in various communities for a long time. At a

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time when metal was hard to come by, wood was used as an alternative. It was light, easy to attain, easy to work with, and it had its own strength when it came to construction work and joints. Wood has played an important part in both old and modern materials, proving easy to mold into various shapes and connecting parts together.

It's expected for forces to act upon various pieces of construction, thus, to ensure the durability of the object, certain measures have to be taken. For example, the accurate placement of the dowel hole, its size, and the overall tightness. All are necessary so that the wooden dowel wouldn't be weakened under such force.

The idea was given by the company (OÜ Natur) who makes different kinds of wooden chairs, especially made out of birch. To connect different details in a chair the company use wooden dowels. In the chair construction, it is very important that the cross rails can withstand different kinds of forces. This is why the company has taken to use twisted wooden dowels, because the technology of making twisted dowels is based on by pressing those rails. The company was interested in what kind of dowel is better in tensile and bending strength and also how much glue was necessary.

## MATERIAL AND METHODS

The aim of the experiment is to find out the strength of both the twisted birch dowel and the fluted birch dowel. In order to gain accurate and precise results, the author tested, using one dowel at a time, both the fluted and the twisted dowels.

As there were no current standards for the experiment, the author, using any equipment available to him, made his own method of testing, using IMAL IB 600 (Fig. 1). The first thing was making figures of the test blocks. The materials and machines were given by OÜ Ami Treipuit, a company that also used birch to construct details.



Figure 1: Test machine IMAL IB 600

## Drilling holes

When the blocks were ready, the next step was to drill holes. Several tests were done on varying lengths of dowels, using two different diameters, 8mm and 10mm.

In order to connect details using wooden dowels, it is recommendable that the hole in the top of the detail is supposed to be 0.6 of the dowel length and the hole in the side of the detail is 0.4 of the dowel length. A metal caliber was used to control.

## Gluing the blocks

The glue used in the tests was a polyvinyl acetate, *DORUS DD 060*, characterized as having a high viscosity and recommended specifically for wooden dowels.

The combination of the glue and wooden dowel help strengthen the bond between the two details, against the forces of bending and tensile.

In order to glue the test pieces, the author had to follow a list of conditions set by the brand of glue, *DORUS DD 060*.

Room temperature, °C	18-23
The room relative humidity, %	40-60
Moisture content of wood, %	8±2
Shelf life of the glue, months	12

To be sure about the glue viscose, the author controlled that by using the viscometer VISCO STAR L (Fig. 2). The viscose of the glue was 294 mPa s.



Figure 2: Viscometer VISCO STAR L

## Tools used for gluing

- Syringe – Used to place the glue with the same sized drops
- Pencil – Used to spread the glue around in the dowel hole
- Toothbrush - Used to spread the glue around the dowel
- Plastic Film – Placed between the blocks in order to stop the details from gluing to each other
- Hammer – Used to strike the dowel
- Calliper – To measure the dowel

After trial and error, the author figured out the perfect amount of glue to be placed in the dowel hole (Fig. 3. and 4.).

Using a dowel with a diameter of 8 mm and length of 40 mm, the amount for the hole was 0.47 g (12 drops).

Using a dowel with a diameter of 10 mm and length of 40 mm, the amount for the hole was 0.8 g (18 drops).

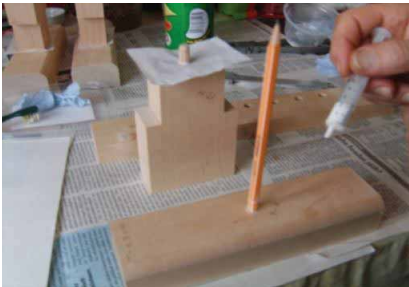


Figure 3: Gluing tensile test block



Figure 4: Gluing bending test block

## RESULTS

### Testing tensile and bending strength

For tensile strength, the tests show resistance in Newtons between fluted and twisted dowels and the wood (Fig. 5.). To get those results from the computer, the author calculated the tensile strength for the glued surface in  $\text{N}/\text{mm}^2$ . Bending strength is very important to calculate for different kinds of wooden joints. The tests show the exact resistance to compare between twisted and fluted dowels (Fig. 6.). The test results are shown in Table 1. and Table 2.

To compare the diameters of the dowels, the author used a 3D scanner. For the fluted dowel, there were some technical mistakes.





Figure 5: Testing tensile strength



Figure 6: Testing bending strength

Table 1: Tensile strength (P) and glued surface (Q) in dowel with diameter 8 mm

Test No.	Dowel type	Hole depth		P, N	Q, N/mm <sup>2</sup>
		By the side	On the top		
1	Twisted Ø 7.9 x 40 mm	21 mm	21 mm	3076	6.2
2	Twisted Ø 7.9 x 40 mm	21 mm	21 mm	3540	7.13
1	Twisted Ø 8 x 40 mm	21 mm	21 mm	3186	6.34
2	Twisted Ø 8 x 40 mm	21 mm	21 mm	3630	7.23
1	Fluted Ø 8 x 40 mm	21 mm	21 mm	3941	7.84
2	Fluted Ø 8 x 40 mm	21 mm	21 mm	3596	7.16
1	Twisted Ø 7.9 x 40 mm	16 mm	16 mm	3606	9.69
2	Twisted Ø 7.9 x 40 mm	16 mm	16 mm	2657	7.14
1	Twisted Ø 8 x 40 mm	16 mm	16 mm	2774	7.36
2	Twisted Ø 8 x 40 mm	16 mm	16 mm	3150	8.36
1	Fluted Ø 8 x 40 mm	16 mm	16 mm	4057	10.77
2	Fluted Ø 8 x 40 mm	16 mm	16 mm	3605	9.57

Table 2: Bending strength (P) and glued surface (Q) in dowel with diameter 8 mm

Test No.	Dowel type	Hole depth		P N	Q, N/mm <sup>2</sup>
		By the side	On the top		
1	Twisted Ø 7.9 x 40 mm	21 mm	21 mm	1529	31.21
2	Twisted Ø 7.9 x 40 mm	21 mm	21 mm	1506	30.74
1	Twisted Ø 8 x 40 mm	21 mm	21 mm	1547	30.79
2	Twisted Ø 8 x 40 mm	21 mm	21 mm	1935	38.52
1	Fluted Ø 8 x 40 mm	21 mm	21 mm	1981	39.43
2	Fluted Ø 8 x 40 mm	21 mm	21 mm	1547	30.79
1	Twisted Ø 7.9 x 40 mm	16 mm	16 mm	1683	34.35
1	Twisted Ø 8 x 40 mm	16 mm	16 mm	1579	31.43
1	Fluted Ø 8 x 40 mm	16 mm	16 mm	1309	26.05

## CONCLUSION

In this experiment the main purpose was to find differences in tensile and bending strength between the twisted and fluted wooden birch dowels. The first thing that had to be worked out was the method to follow.

### **Tensile strength**

In carrying out the tensile tests, it showed that the fluted dowels could withstand more force. All the conditions needed for the experiment stayed the same throughout the tests. While it is important that the plug hole diameter corresponds exactly to the dowel diameter.

Results of the tests of the surface bending strength  $N / mm^2$  for the fluted dowel was in the range of 5.19 to 11.16  $N / mm^2$  (MPa), compared to the twisted dowel, where the results ranged from 6.22 to 10.77  $N / mm^2$  (MPa).

### **Bending strength**

The testing was to find the shear strength of the dowel. The test results showed that the twisted dowel was up to 10% stronger than the fluted dowel. In the manufacture of twisted dowels, it is pressed down to the appropriate size, thus getting denser and having a greater resistance than the fluted dowel.

During the tests, a 40 mm long dowel was used, and the testing blocks each had a hole depth of 21 mm. As force was applied, the author observed the dowel break, as well as crush the hole of the vertical test block.

To have stronger joints:

- Use twisted dowels
- Increase the diameter of the dowel plugs
- Increase the number of dowels (3 or more) if the structure allows. This solution increases the shear stability.

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# MODELLING STIFFNESS AND HARDNESS VARIATIONS IN LITHUANIAN GROWN OAK

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## ABSTRACT

This paper discusses the first phase of a project focused on performance and indications of possible enhanced mechanical properties for the treated compared with untreated Lithuanian-grown oak wood. The aim of the present study was to examine visco-elastic performance of untreated oak specimens intended for flooring products. Specially selected, sampled and grouped accordingly to growth characteristics and physical properties oak (*Quercus*) specimens with 28 mm thickness, 80 mm width and 625 mm length were characterized by mechanical testing and linking dynamic modulus of elasticity, Brinell's hardness and main physical wood properties. The density, sound of velocity, ultrasound frequency, dynamic modulus of elasticity (MOE) and Brinell's hardness (HB) modulus were measured. A high correlation was observed between wood dynamic modulus of elasticity MOE and density, Brinell's hardness HB and density and between MOE, HB and density. For better understanding applied character of oak wood properties experiments were also configured accordingly to the relevant European Standards.

Key words: sound of velocity, density, dynamic modulus of elasticity, Brinell's hardness.

## INTRODUCTION

A large number of studies concerning the strength properties of softwood have been conducted because the structural members in timber constructions are composed mostly of softwood [1]. The mechanical properties of some hardwood species that are used as finishing materials and in the manufacture of furniture and flooring also need

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to be clarified [2]. Hardness of wood has a good relation with its various mechanical properties [3]. For this reason, hardness is important as one of the main indices of wood quality [4].

For wood properties investigations are exploited X-ray (for searching of internal defects) [5], infrared ray tomography [6], ultrasound investigations [7-9], etc. Wood is biological material, so it characterized as variations of physical properties and directional fibred structure – anisotropy. Long-time wood properties were investigated by destructive methods, such as compression perpendicular/parallel to grain, tension perpendicular/parallel to grain, bending strength etc. [10]. When these methods are applied samples are destroyed and not useable for the further applications. Because of increasing costs of timber and decreasing wood resources such methods are not valuable. So recently non-destructive test methods are increasingly used in research and industry, particularly for quality control in the manufacturing of wood products [7].

Accurate identification of mechanical properties of wood material without altering its end-use capability is the basis for nondestructive evaluation (NDE). Industrially, NDE has also been used as a quality control tool in the manufacture of wood-based materials. The time taken to conduct a NDE is shorter than the alternative attic test. The use NDE can provide knowledge of the properties of wood. Dynamic mechanical analysis of wood has been shown to be useful in predicting modulus of elasticity (MOE) and decay. It is more difficult to predict modulus of rupture (MOR) due to the presence and location of defects and slope of grain of wood that have significant effect on these properties [11].

Ultrasound investigations of solid wood are usably about 20 years and still have limited use in the industry. They are applicable in the micro scale as well at macro level starting from standing trees, logs to sawn timber for searching internal defects (knots, cracks, rots, etc.) or to determine directly modulus of elasticity and predict modulus of rupture (strength properties) [12].

Characteristics such as time-of-flight, free vibration, wave frequency and wave velocity are successfully used to evaluate properties of wood materials [13]. New methods and equipment are seeking applications for non-destructive methods of wood investigation. Basic types of waves when investigated wood properties are bulk (volumetric) and surface waves. Influencing factors for wave traveling through the wood are: physical properties of specimen, geometrical features (micro and macro structure), inner layer conditions (temperature and moisture content) and measurement equipment [12].

The vibration properties of primary interest in structural materials are speed of sound and internal friction (damping capacity). The speed of sound in a structural material is a function of the modulus of elasticity and density. In wood the speed of sound also varies with grain direction because the transverse modulus of elasticity is much less than the longitudinal value (as little as 1/20); the speed of sound across the grain is about one-fifth to one-third of the longitudinal value [14].

The speed of sound decreases with increasing temperature or moisture content in proportion to the influence of these variables on modulus of elasticity and density. The speed of sound decreases slightly with increasing frequency and amplitude of vibration, although for most common applications this effect is too small to be significant. Variability in the speed of sound in wood is directly related to the variability of modulus of elasticity and density [14].

The principal use of hardwood lumber is for remanufacture into furniture, cabinetwork, and pallets, or direct use as flooring, paneling, molding, and millwork. Flooring is exclusive finished market product with the high production volumes worldwide [14].

The aim of this study was to propose quality indexes to evaluate the dynamic modulus of elasticity and hardness that are essential properties of various hardwood products. We focused on the linking properties of the density, annual rings width, and grain angle in crosscut surface with the specimen hardness and stiffness. In order to propose quality indexes we measured various mechanical properties and discuss the relationships between these properties.

In this research work were used acoustic measurements of sound velocity in the oak wood specimens for estimation of dynamic modulus of elasticity (DMOE). Applied Lamb waves travel along the length through the whole specimen volume if maximum depth does not exceed 80 mm. Applicability of acoustic measurement method was estimated after results analyzing.

## MATERIAL AND METHODS

Physical-mechanical properties of initial 234 oak samples were evaluated and 110 test specimens selected for final testing and grouped considering density and grain directions: across the grain, parallel to grain and perpendicular to grain. Density groups were divided as follows: 550-600, 600-650, 650-700, 700-750, 750-800 kg/m<sup>3</sup>. For all specimens were measured moisture content, average width of annual rings, and grain inclination in the crosscut.

Average specimens dimensions were: length – 625 mm, width – 80 mm and thickness – 28 mm. They were conditioned at 20° C and 65 % humidity. Average moisture content during testing varied at 7-13 %, average sample density recalculated to 12 % moisture content was 737 kg/m<sup>3</sup>.

Acoustic investigations were done with electronic equipment, made at the Institute of Metrology of Kaunas University of Technology. The longitudinal stress wave method used in this experiment based on the Lamb stress wave propagation principle. It uses the velocity at which an induced wave travels from the sender (starter) point sensor to the receiver (stopper) point sensor along the 370 mm length of the tested material. After placing sensors on the tested oak timber specimen the receiver fix and display the

time spent on such length. Lamb waves travel throughout all specimen depth. In each specimen wave traveling time was measured 5 times at the same points and average of the time spent by the wave signal was calculated. The wave velocity is calculated by formula:

$$C = \frac{L}{t \cdot 10^{-6}}, \text{ m/s} \quad (1)$$

where  $L$  – distance from sender and receiver (370 mm),  $t$  – the time spent by the wave signal ( $\mu\text{s}$ ).

The dynamic modulus of elasticity (DMOE)  $E_D$  was calculated by formula:

$$E_D = C^2 \cdot \rho, \text{ N/mm}^2 \quad (2)$$

where  $\rho$  - density of the wood,  $\text{kg/m}^3$ .

Hardness was calculated according EN 1534 standard [15]. Indentations shall be evenly distributed over the face area of the element. The test specimen was set on the table of the testing machine to lower loading head with the indenter to the surface of the test specimen. Indenter is hardened steel spherical body with contact diameter of  $(10 \pm 0.01)$  mm. Increasing force was applied at such rate that the nominal value of 1 kN is reached after  $(15 \pm 3)$  s. The force was maintained at this value for  $(25 \pm 5)$  s. After withdrawal of the indenter wood was let recover for at least 3 min. Two diameters were measured along and across the grain with the accuracy of  $\pm 2\%$  and calculated average of them.

The Brinell hardness  $HB$  was calculated according to the following formula:

$$HB = \frac{2F}{g \cdot \pi \cdot D \left( D - \sqrt{D^2 - d^2} \right)}, \text{ N/mm}^2 \quad (3)$$

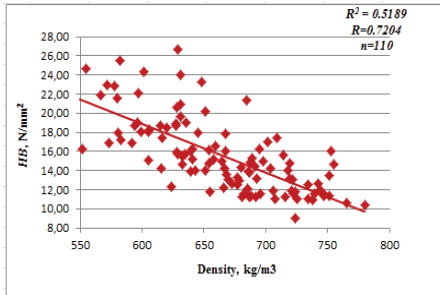
where  $g$  – the acceleration of gravity ( $9.8 \text{ m/s}^2$ );  $\pi$  - „pi“ factor (3.14);  $F$  – maximum load applied force, N;  $D$  – the diameter of the ball, mm;  $d$  – average diameter of residual indentation, mm.

Statistical processing was made using program software „STATISTICA v.10“. Linear and non-linear regressions for best modeling adequacy and validation were applied.

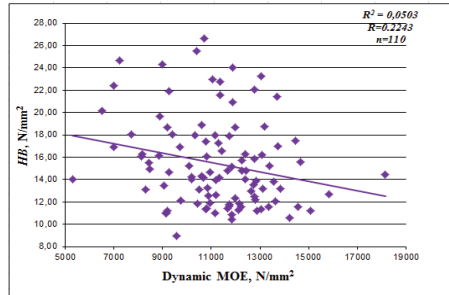
## RESULTS AND DISCUSSIONS

Initial testing of ultrasonic wave propagation time-of-flight was focused on evaluation Lamb wave efficiency to detect and measure internal wood structure and inhomogeneity when placing KTU stiffness tester on each specimen plane, e.g., both edges and sides. Statistical processing of 20 times repeated on each specimen plane measurements showed minimal not exceeding 2.1% dispersions of sound velocity values received when locating tester on particular planes. Therefore, acoustic Lamb wave traveling through the wood distinguishes by exceptional bulk (volumetric)

propagation ensuring spatial inspection of wood internal structure and inhomogeneity. Further testing of all specimens and processing of measurement results and recalculation of time-of-flight into elasticity characteristics showed variation of dynamic MOE for all specimens at 1222 N/mm<sup>2</sup>, and variation of natural frequency at 5430 Hz. Average sound velocity varied at 4281 m/s. Parallel assessments of Brinell's hardness resulted in hardness variation for all specimens at 12,6 N/mm<sup>2</sup>. Rigorous variations of hardness and stiffness are shown on Figures 1 and 2.

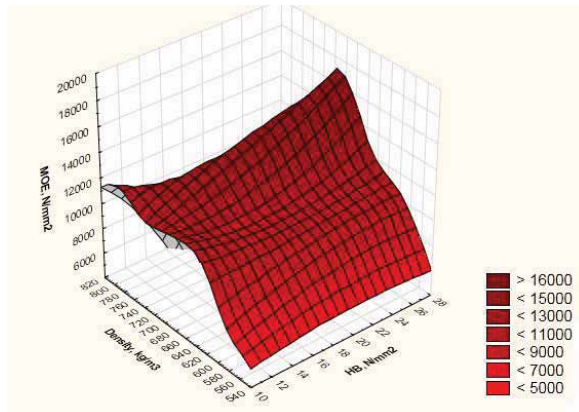


**Fig.1.** Dependence of Brinell's hardness from density of selected 110 oak wood specimens



**Fig.2.** Relation of Brinell hardness and dynamic MOE of selected 110 oak wood specimens

Relation between Brinell's hardness and density is confident (Fig. 2) while prediction of hardness by dynamic MOE (and vica-versa) is unreliable ( $R^2=0.0503$ ). But adding density as a modelling factor makes such interrelation much more calculable (Fig. 3 and formula 4).



**Fig.3.** Dependence of dynamic MOE from Brinell's hardness and density of 110 oak wood specimens.

Linear regression equation for this model is:

$$E_D = -3947.49 + 89.3 HB + 20.28 \rho, \text{ N/mm}^2 \quad (4)$$

Coefficient of determination for this model is  $R^2=0.185$ , coefficient of correlation is  $R=0.43$ .

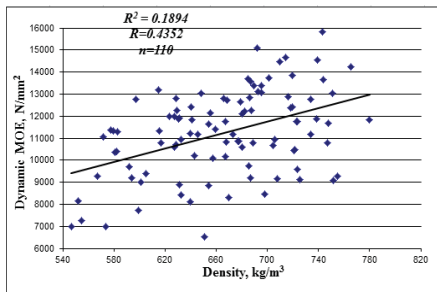
Non-linear equation gives even better for MOE prediction model:

$$E_D = -4725.11 - 5.6 HB + 303.5 \rho - 0.1 HB^2 + 136.3 \rho^2, \text{ N/mm}^2 \quad (5)$$

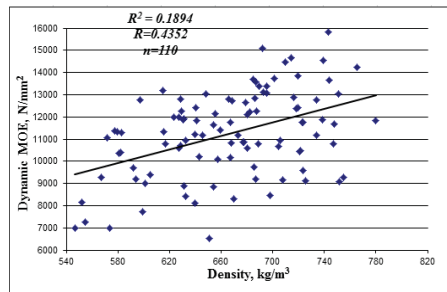
Coefficient of determination for this model is  $R^2=0.2099$ , coefficient of correlation is  $R=0.4581$ .

Similar modelling is well known and effective for performance prediction of structural members [16, 17] and our intentions here were to discover easy available but sufficiently confident parameters for further simplified multifactorial modelling of hardness and stiffness-strength properties.

Examining elastic wood properties using Lamb waves gives typical for ultrasonic methods prediction of dynamic stiffness on wood density (Fig. 4 and 5). In general received characteristics are in line with the classical knowledge [7, 8] but we conclude slightly higher than usual [9] MOE dispersion of received results (7000-16000 MPa) for randomly selected Lithuanian-grown oak specimens (Fig. 4).

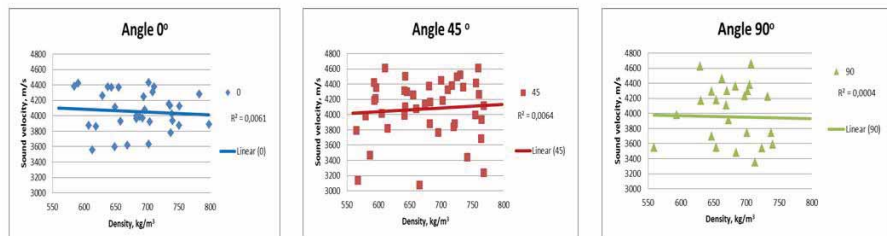


**Fig.4.** Dependence of dynamic MOE from density for all 234 oak specimens



**Fig.5.** Dependence of dynamic MOE from density for selected 110 oak specimens

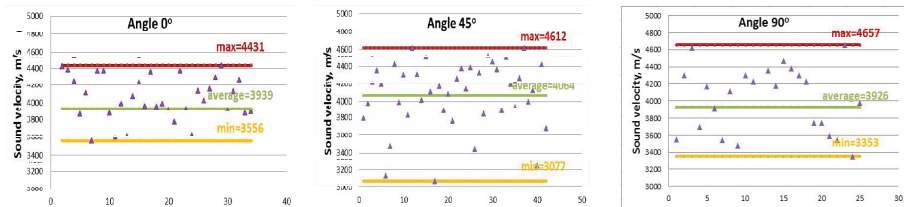
Density well explain MOE and HB variations but for applied structural use of load bearing members stiffness and hardness prediction from density is not sufficient. Modelling MOE with density and hardness (model 4) is usable but with limited accuracy. We examined cross-sectional grain inclination as an additional modelling factor and it proved to be successful for enhancing models reliability and verification. (Figures 6 and 7).



**Fig. 6.** Dependence of sound of velocity from grain angle



Radial, tangential and 45 degree specimens do not expose notable tendencies on changing ultrasound velocity for various densities (Fig. 6 and 7). At 0° and 90° degrees for higher wood densities ultrasound velocity slightly decreases while at 45° it shows little growth (Fig. 6).



**Fig. 7.** Distribution of sound velocity of specimens cut with different grain angle. Yellow line is min value, green line is average value and red line is maximum value

As seen on Fig. 7 main problem cause enormous variation of sound velocity but average values remain surprisingly similar for tangential, radial and 45° annual ring specimens. Dependence of dynamic MOE can be described by equation:

$$E_{dynamic} = -18237.3 + 4.8C - 0.4Angle + 3.8HB + 14.9\rho \quad (6)$$

Coefficient of determination for this model is  $R^2=0.9401$ , coefficient of correlation is  $R=0.9696$ . Model 6 gives reliable and confident prediction of MOE based on specimen density, hardness and ring angle at the specimen crosscut. Together with the equations 4 and 5 it makes basis for understanding and modelling wood viscous-elastic properties and simplified wood characterization at untreated conditions but most effective applications can be received for fast-track monitoring of dynamically changing properties during wood thermo-chemical modifications.

## ACKNOWLEDGMENTS

Lithuanian Science Council through the European Cooperation Program in the Field of Scientific and Technical Research (COST Action FP 0802) supported this work.

## CONCLUSIONS

1. Acoustic Lamb wave traveling through the wood distinguishes by exceptional bulk (volumetric) propagation ensuring spatial inspection of wood internal structure and inhomogeneity.
2. Lamb wave time-of-flight, wave frequency and wave velocity are efficient tools to evaluate and model stiffness-strength and hardness properties of wood materials.
3. Annual ring inclination in cross-section along with wood density proved to be successful for enhancing wood stiffness-hardness models consistency and reliability.

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# NUMERICAL MODELLING AND EXPERIMENTAL VALIDATION OF DENDROLIGHT® CELLULAR WOOD MATERIAL

Labans, E.<sup>1</sup> & Kalnins, K.<sup>2</sup>

## ABSTRACT

The aim of a current research is to estimate most appropriate numerical modelling approach in order to predict mechanical behaviour of small scale DendroLight® specimens under compression/bending loading conditions. For this task several finite element (FE) modelling techniques has been utilised assessing the supremacy of shell our solid finite element models in ANSYS and ABAQUS commercial software. Due to complicated DendroLight® structure topology a special attention has been devoted to experimental validation of numerical models. Several series of DendroLight® cellular specimens have been tested in longitudinal and transverse compression along with four point bending sandwich samples with HDF (High Density Fibreboard) skins. It has been confirmed that both finite element model types predict specimen mechanical behaviour sufficiently well compared to results obtained experimentally. Obtained differences usually not exceed 20% margin. Moreover it has been concluded that solid elements are more appropriate as they delivers more extensive information about the stress/strain distribution compared to the shell element model.

Key words: DendroLight®, cellular wood structure, finite element modelling, mechanical testing, ANSYS, ABAQUS

## INTRODUCTION

DendroLight® is a unique wood material specifically developed as core material for furniture industry. Manufacturing started at the year 2010 in Ventspils, Latvia. It is made from profiled/perforated wood boards stacked in perpendicular layers and then sliced once more in plates perpendicularly to the board's layers. The main advantage of such a solution is significant reduction of structural weight (up to 40 %) comparing to

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conventional timber. Thus such a cellular wood material has a potential to be utilised in near future in load bearing structures, like walls and floors as core of sandwich panels. For this task a reliable structural analysis methodology is required, able to assess detailed geometry component (like profiled board web thickness) influence over the stiffness for large scale structure.

Computer software based on finite element method has invaluable influence over modern engineering design practice in various steps from design of a wood furniture pieces up to building structures. Computer analysis allows achieving higher accuracy and reducing the calculation time for complicated models comparing with the analytical solution. The finite element analysis becoming more exploited for conducting research related to development of innovative wood products. For example recently Persson (2008) devoted his doctoral thesis for elaborating of mechanical behaviour of the wood cell walls using an ABAQUS code. Moreover the finite element analysis has been acknowledged by number of researchers as convenient method for analysing stress/strain distribution in wood connections with the steel fasteners (Nishiyama and Ando 2003, Resch and Kaliske 2010). Moreover Zhou et al (2010) analysed seismic behaviour of the wood structures employing ANSYS code. A most comprehensive summary is given by Mackerle (2005) in review article regarding the wood product development and researches with emphasis to FEM application.

In case of DendroLight<sup>®</sup> the ANSYS (2009) and the ABAQUS (2009) software were utilised for simulation of mechanical behaviour of cellular wood structure under compression and bending loading conditions. DendroLight<sup>®</sup> has not yet been widely investigated. A basic set of material properties are described by manufacturer (2012) and Iejavs et al (2011) has conducted experimental investigation on a large scale wood sandwich panels and concluded that the cellular wood material could be successfully applied as the core material.

In this particular paper a comparison of different FE techniques for modelling of DendroLight<sup>®</sup> structure has been given. It has been evaluated the time consumption for model creation, meshing efficiency and stress/strain information amount acquired from the model. To assess the model accuracy numerical results were compared with the values obtained in experimental tests.

## MATERIALS AND METHODS

### FEM modelling

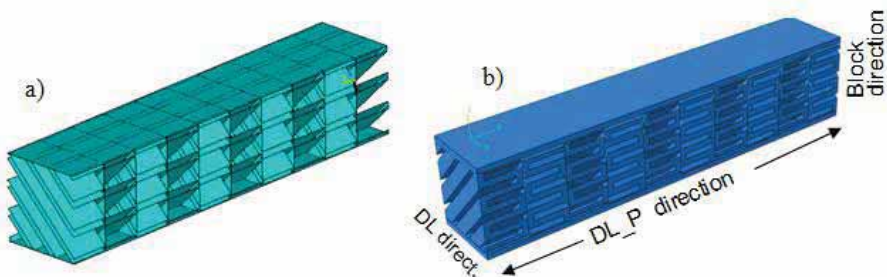
Mechanical properties employed for modelling of wood material are as follows: The modulus of elasticity in longitudinal direction - 11 GPa; the modulus of elasticity in transverse and radial direction  $E_T = E_R = 0.6$  GPa, the shear modulus:  $G_{LR} = G_{LT} = 0.34$  GPa,  $G_{RT} = 0.2$  GPa and aPoisson's ratios  $\mu_{LR} = \mu_{LT} = 0.34$ ,  $\mu_{RT} = 0.03$ . The Young's modulus is taken according to the timber strength class C24 in European standard EN338 (2003). Other properties evaluated using the Wood handbook (1999). Such

properties are more characteristic for pine, however accounting influence of the wood species was out-of-scope in current research, because manufacturing technology currently is set to utilise both pine and spruce raw material.

Corresponding isotropic mechanical properties have been assigned also to High Density Fibreboard (HDF) skins:  $E_{HDF}=3.2$  GPa,  $\mu_{HDF}=0.3$ .

Numerical model in the ANSYS software has been made employing SHELL181 elements with transversal isotropic wood material properties (Figure 1.a). Elements were connected using node-to-node connections at coincident points. The cellular wood structure created in sequence of real production process, starting with profiled board modelling, forming layers and cutting blocks into DendroLight<sup>®</sup> layers. Boundary and loading conditions have been set according to experimental test set up. The ABAQUS software has been used for modelling DendroLight<sup>®</sup> structure from solid type elements. Due of prismatic component forms mainly tetrahedral element shapes have been developed in meshing process. Profiled boards and cellular wood core surfaces have been connected together with surface-to-surface bounding. Mesh size step were set to magnitude of 8 mm. Largest of modelled specimens *B2* (see details in Table 1) has about 12000 shell elements in ANSYS and 15700 solid elements in ABAQUS.

Structural loads were assigned to sets of nodes with jointed deflections along vertical direction. It allows simulating uniform pressure on top plate in case of compression specimens and line loads with rollers in case of bending specimens. Boundary conditions for compression specimens have been assigned by selecting all lower nodes and restricting their translations along all axes. In case of bending specimens boundary conditions have been applied only at the ends of the sandwich beam. Bounding conditions allows the rotation of all nodes and translation of nodes along longitudinal direction for one end of the beam type specimen.



**Fig.1.** Finite element models for sandwich beam specimens a- ANSYS shell 281 model, b-ABAQUS – solid 3D model






In order to reduce the calculation effort only the linear analysis has been performed. Such an approach is in line with good design practice, where the serviceability limit state is reached much faster than the ultimate limit state.

## Experimental investigation

To assure created numerical model accuracy a several test series for the small scale specimens have been performed. Specimens have been tested in compression and bending in ZWICK Z100 testing equipment (Figure 2). All specimens for bending set-up have 4 mm thick HDF skins. More detailed specification of tested specimens is given in Table 1. It has been assumed that profiled board thickness for all specimens is kept constant 25 mm.

Specimens have been loaded until failure in quasi static compression with the test speed of 1 mm/min. Displacements have been measured by the machine crosshead travel. Mechanical properties of DendroLight<sup>®</sup> largely depend on wood cell direction; therefore specimens with different orientations have been evaluated (Directions are shown in Figure 1.b). For compression specimens mechanical properties are different in two directions namely: 'DL' and 'Block'. 'Block' direction for compressed specimen and 'DL' direction for bending sandwich specimen is displayed in Figure 2. For bending specimens are possible three types of core orientation affecting mechanical properties. Those similar to compression specimens and additionally 'DL\_P' which is 'DL' direction turned perpendicularly to bending the specimen longitudinal direction.

**Table 1** Specification of tested specimens

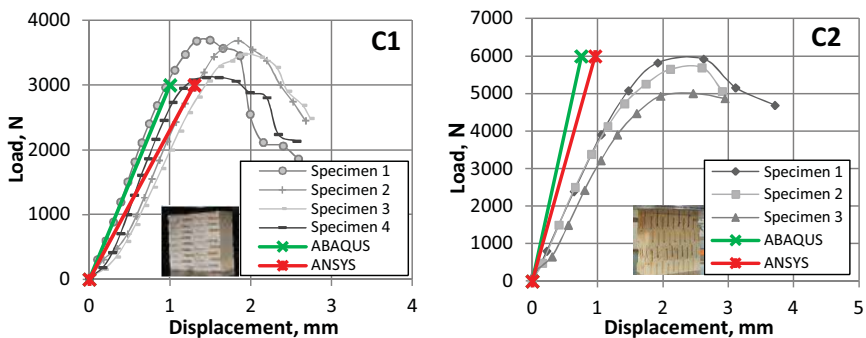
Notation	Dimensions			Number of specimens	Test mode	Structure orientation
	Length, $L$ [mm]	Width, $B$ [mm]	Height, $H$ [mm]			
$C1$	100	40	100	4	Compression	DL 
$C2$	100	40	100	3	Compression	Block 
$B1$	200	50	40	2	4-point bending	DL 
$B2$	300	50	60	3	4-point bending	DL_P 
$B3$	350	50	30	3	3-point bending	DL 



**Fig.2.** 3- point bending and compression (on the right) test set-up on ZWICK Z100

## RESULTS AND DISCUSSIONS

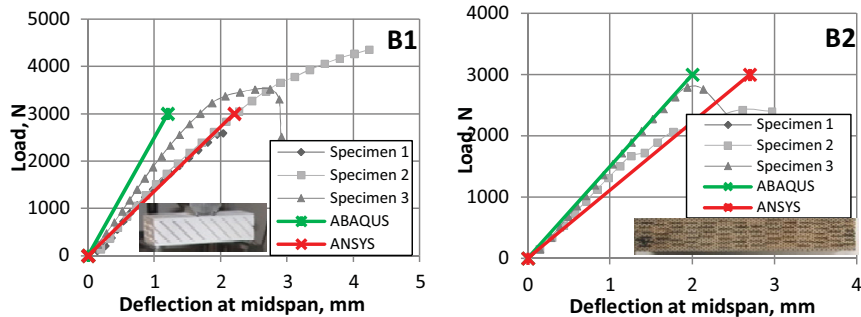
In order to ease the comparison of experimental and numerical results a load deformation curve plot has been elaborated. One may see that the experimental results for compression specimens are shown in Figure 3. An average Modulus of elasticity for *C1* series specimens is near 50 MPa, for *C2* series specimens 110 MPa (calculated using EN789 (1995) methodology for reduced specimen size). Specimens have clear elastic mechanical behaviour zone until 80 % of critical load. It makes reasonable use of linear numerical model. Numerical results are in good agreement with *C1* series specimens, however for *C2* type specimens numerical results shows more than 40 % less stiffness. It leads to assumption that wood mechanical properties in radial direction are weaker than applied in numerical model. Long plasticity region for *C2* specimens appears due to slow buckling and crushing of profiled board walls. Comparing to *C1* series specimens there is no rapid losing of load carrying capacity.



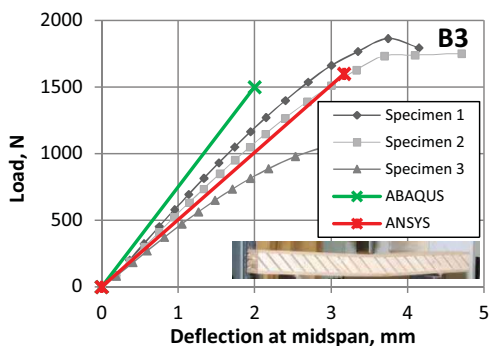
**Fig.3.** Experimentally obtained displacement values compared with numerical results for compression specimens *C1* and *C2*

Mechanical behaviour of bending specimens mainly depends on properties of outer skins. As in previous plots all specimens have clearly visible elastic behaviour region. Except for *B2* specimen's elastic region is only half of the critical load due to appearance of shear deformations when bond between profiled boards was lost near the

loading point. Numerical results in linear mechanical behaviour regions are close to experimental values. In general ABAQUS numerical model has higher stiffness than model from shell elements.



**Fig.4.** Experimentally obtained deflection values compared with numerical results for bending specimens *B1* and *B2*



**Fig.5.** Experimentally obtained deflection values compared with numerical results for bending specimens *B3*

Comparing calculation times on a single PC with two core processor it is obvious that model from shell elements (made in ANSYS code) has shorter analysis time comparing to ABAQUS model made with solids. For *B2* specimen in case of shell model – 20 seconds; for solids model - 140 seconds. Calculation time could be especially important for further optimisation tasks of sandwich panels topology, where several hundreds of experimental runs are required on the full scale structure. Model in ANSYS is made fully parametrical therefore it is more computational time efficient for creating of model variations. In case of *B3* specimen it takes nearly 280 seconds for analysis task. Solid geometry for ABAQUS was made employing SOLIDWORKS CAD software and then imported for analysis. Both codes have a good compatibility because are made by the same developer company. However to make parametrical ABAQUS model, programmable features in Python language should be elaborated



instead of SOLIDWORKS 3D drawings. Another challenge for numerical modelling is appearance of the small mesh elements during geometry forming operations. They could dramatically decrease calculation speed or in worst case scenario to cause crash of the calculation process entirely.

## CONCLUSIONS

It has been demonstrated that by employing numerical modelling it is possible to predict mechanical properties of DendroLight<sup>®</sup> sandwich panels with sufficient accuracy in elastic deformation range. For experimentally tested specimens the margin is up to 80% of critical load. The obtained differences between numerical and experimental results usually do not exceed 20 %. In most validation cases numerical models made by solid elements in ABAQUS code demonstrated higher stiffness than model of shell elements made by ANSYS code. Nevertheless a shorter calculation time is possible to reach by creating the numerical model from shell type elements.

Taking into the account relatively long calculation time for creating and analysis of detailed DendroLight<sup>®</sup> structure, such an analysis is suggested only for small scale structural elements with dimensions not exceeding one meter of length. For large scale structures mixed approach should be utilised instead when mechanical properties are extracted from small scale numerical model and assigned to continuum layer of large structure as equivalent stiffness properties.

Further investigation is required to improve numerical model's accuracy also including adhesive layer and structural imperfections as well to decrease the calculation time and to improve the model calculation stability.

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# IMPACT OF TECHNOLOGICAL FACTORS ON THE ROUGHNESS OF MILLED WOOD SURFACE

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## ABSTRACT

This article presents the research results showing wood surface roughness changing when milling with variable cutting path of milling tool, feed and cutting speed. The tests performed with the wood samples of birch and pine. The specimens were milled along the fiber in the experimental wood cutting stand at two different cutting and two feed speeds. The roughness parameter  $R_z$  of the processed specimens was measured in five sectors using the contact stylus surface roughness measuring instrument. The roughness was measured in each sector along and across the fiber and received measurement results were processed by Gaussian digital filter. The statistical analysis ranked machining surface roughness parameters by diminishing significance as feed speed, tool nose cutting path and peripheral cutting speed.

Key words: wood milling, surface roughness, birch wood, pine wood

## INTRODUCTION

Cutting is such a technological process that destroys links between the particles of processed material accordingly to the predicted surface in order to receive a product of specific dimensions, spatial form and surface roughness. During the technological process the tool gets blunt, and thus the roughness of the surface being processed changes. The surface roughness is the complex of microgeometrical unevenness on the surface, when the micro irregularities forming the profile of the surface repeat by some small step. The roughness determines further processing and finishing mode and usage possibilities. The accuracy of geometrical parameters is one of the main tasks, when the wooden products are being made (Koch 1964, Lubchenko 1986, Baltrušaitis 2009).

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Wood possesses specific structure and mechanical processing of wood is highly affected by natural anisotropy. The anatomic unevenness depend on the biological species of wood, its density, moisture content, number of annual rings per 1 cm, and proportion of early and late wood in the annual ring. The softwood and hardwood have different modes of anatomic surface unevenness. Besides, the anatomic unevenness dramatically depends on the direction of wood fiber. The wood cells, which make the anatomic unevenness of wood surface, are cut or deformed during the mechanical processing. Therefore the unevenness, which dimensions resemble those of the cell, is formed. When the mechanical wood processing is analyzed, the anatomic unevenness usually is not taken into account. Usually they are considered as small if compared with the kinematic unevenness caused by mechanical processing (Fujiwara *et al.* 2004, Benkova and Schweingruber 2004, Jakimavičius 2008).

The kinematic unevenness is formed due to the peculiarities of mechanical processing. The wood processing process causes cutting forces, vibrations, emission of heat, electrochemical phenomena and other factors. Each factor affects the technological process and predetermines unevenness on the surface. The size of unevenness depends on the processing mode (cutting, planing, filing, drilling, sanding etc.), microgeometry of the tool blade cutting edge (nose), cutting direction, and of the cutting and feed speeds. The vibratory unevenness appear because of vibrations of tool or blank, and irregular movements of cutting and feed speeds (Jackson *et al.* 2002, Ohtani *et al.* 2004, Keturakis and Juodeikienė 2007, Keturakis and Jakubauskaitė 2009, Hiziroglu and Kosonkorn 2006, Kiliç *et al.* 2006, Malkoçoğlu 2007, Aslan *et al.* 2008).

The surface roughness is measured using various measuring tools, which operate by the non-contact, contact and subjective measuring methods. The advantage of devices working by non-contact method is that they do not touch the measured surface, do not contact with it, and do not cause mechanical deformations or other damages. The optical and interference devices are attributed to non-contact measurement devices (Sandak *et al.* 2004, Sandak and Tanaka 2005).

The devices working by contact method touch the surface being measured by measuring tip (needle or probe), which movements are transferred to the device's scale or recorded using the optical-mechanical, electromechanical or other methods (Sandak *et al.* 2004, Sandak and Tanaka 2005).

The subjective methods are based on visual inspection and comparison of samples with standards. The primary surface roughness is evaluated by human sense. When the wood surface is touched or looked at, it is possible to make initial opinion about it. The anatomical structure of wood, characteristics of pores and changes of color often make measurements of surface roughness more difficult (Sandak *et al.* 2004, Sandak and Tanaka 2005).

The surface roughness, according to DIN 4768 and ISO 4287 standards is assessed by many parameters:  $R_a$ ,  $R_q$ ,  $R_{sk}$ ,  $R_{kt}$ ,  $R_y$ ,  $R_{max}$ ,  $R_z$ ,  $S_m$ ,  $R_{pk}$  and  $R_{vk}$  (DIN 4768 1990,

ISO 4287:1997). The measured results are processed by various filters (Fujiwara *et al.* 2004).

The main objective of this research is to determine the influence of wear of milling tool, cutting and feed speeds on the surface roughness when the birch and pine wood is milled along the fiber.

## MATERIAL AND METHODS

The surface roughness of planed products was researched. The tests were done with wood of biological species of birch and pine (Table 1). The average temperature in the testing room was  $t = 19 \pm 2^\circ\text{C}$ , while relative air humidity was  $\varphi = 50 \pm 5\%$ .

The moisture content of samples was measured by electronic hygrometer (Gann Hydromette H35), with measurement precision  $\pm 0.1\%$ . The number of annual rings in 1 cm was determined by counting the rings in the end section perpendicularly to the wood fiber. In order to determine the density of wood, the sections were cut out from each selected wood sample; dimensions were measured with  $\pm 0.01$  mm accuracy by electronic sliding calipers (Würth 715 76 11). When the volume of sections was determined, they were weighed with  $\pm 0.02$  g accuracy by electronic scales (Excell BH-600).

**Table 1.** Physical characteristics of wood

Wood species	Moisture content $\omega, \%$	Number of annual rings per 1 cm	Average width of annual ring, mm	Average density, $\text{kg/m}^3$
Birch ( <i>Betula</i> )	7...9	5.10	1.96	632
Pine ( <i>Pinus sylvestris</i> )	6...8	4.40	2.27	535

The samples were planed by four-side moulding machine (Weinig Powermat 400 LE and Weinig Unimat 2000 Speed), according to the scheme of longitudinal milling, when the directions of vectors of cutting  $v$  and feed  $u$  speeds are opposite to each other. Two wood milling knives ( $80 \times 40 \times 5$  mm) were fastened in the knives' head (PowerLock), which revolutions were  $n = 8000$  and  $12000 \text{ min}^{-1}$ . The knives were made from high-speed steel (HS 18) and covered with resistant to wear cover Leitz Marathon MC. When the blades of milling knives were fixated in the head, they were jointed for multi-cut surface finishing by each knife in the head. The conditions of milling test are presented in the Table 2.

**Table 4.** Milling test conditions

Cutting speed $v$ , m/s	54.4; 62.8	Width of milling $b$ , mm	40...70
Feed speed $u$ , m/min	16; 21	Cutting circle diameter $D$ , mm	100; 130
Feeding per cutter $u_z$ , mm	0.75...1.30	Number of cutting edge $z$ , unit.	2
Depth of milling $h$ , mm	1...2	Cutting angle $\delta$ , degree	60

The shave's thickness  $a$  was changed indirectly by changing the values of feeding per cutter  $u_z$ . The samples were processed by two cutting speeds  $v = 54.4$  and  $62.8$  m/s. The parameter  $R_z$  of processed surface roughness was measured by electronic contact stylus tip profilometer (Mahr MarSurf PS1). The radius of its diamond tip was  $2 \mu\text{m}$ , measuring angle  $90^\circ$ , and anisotropy measuring length was  $17.5$  mm. The surface roughness unevenness was measured in the intervals of cutting path  $L$ :  $10$ ;  $500$ ;  $1000$ ;  $1500$  and  $2000$  m.

Five sectors were selected in one sample ( $17.5 \times 17.5$  mm), where roughness was measured across and along the fiber. In total 1250 measurements were done during the testing series. All the measurement results were processed by Gaussian digital filter (according to DIN EN ISO 11562). The measurement error has not exceeded the values of roughness parameters  $R_z$  by  $\pm 10$  %.

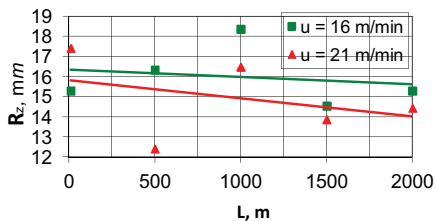
## RESULTS AND DISCUSSION

The researches helped to determine the influence of the wood species, cutting path  $L$ , and cutting  $v$  and feed  $u$  speeds on the roughness of milled surfaces. Besides, it was also determined, how the roughness of milled surfaces changes when measurements across and along the wood fiber are done.

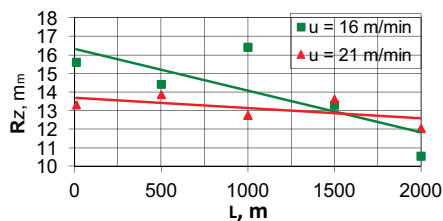
When the measurement results of surface roughness were analyzed, it was noticed that the results are mostly spread in the initial stage of processing. In the first stage the tool suffers mechanical wear through withdrawal and smoothing after-sharpening roughness on the cutting edge. The measurement results of wood surface roughness revealed that the numeric values for birch wood are smaller than those of pine. This regularity is sustained when the surface roughness is measured along and across the wood fiber.

While analyzing the diagrams of results (Fig. 1 a and b), it was determined that when the cutting path  $L$  increases, the quality of processed surface with regard to pine wood is changing saltatory every  $500$  m, i.e. it worsens down to  $8$  % or improves up to  $11$  %. This regularity was noticed at all the feed speeds  $u$ . The smallest surface roughness  $R_z$  is received while milling until the limit of  $500$  m of cutting path  $L$ , when measurements are done along the fiber. When measurements are done across the fiber, the smallest surface roughness is received by the limit of  $1500$  m of cutting path  $L$ . It was quite unexpected that the biggest numeric values of surface roughness  $R_z$  were received when the cutting path was  $L = 1000$  m. The diagrams of results of birch wood (Fig. 2a and b) show that when the cutting path  $L$  increases, the quality of the processed surface improves. This tendency was noticed when the feed speed was  $u = 21$  m/min.

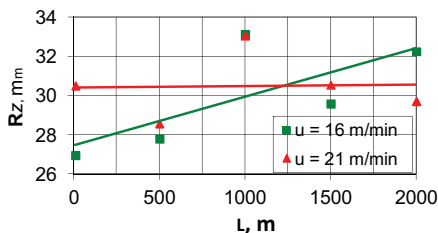
When the feed speed was  $u = 16$  m/min, the quality of the processed surface is not constant when measurements are made every  $500$  m of cutting path  $L$ . It was determined that the  $R_z$  values had increased by average by  $5$  % or decreased by  $6$  %.



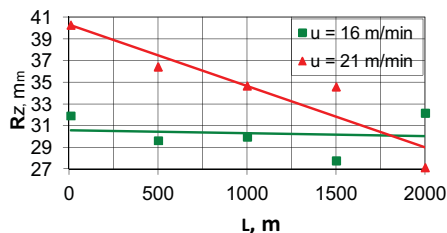
a



a



b



b

**Fig. 1** Surface roughness  $R_z$  of pine wood (when  $v = 54.4$  m/s):

**a** – along the fiber; **b** – across the fiber

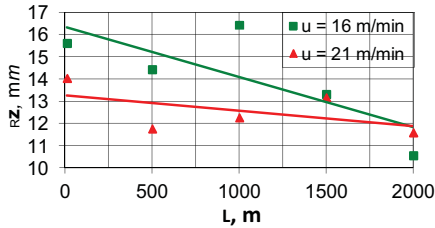
**Fig. 2** Surface roughness  $R_z$  of birch wood (when  $v = 54.4$  m/s):

**a** – along the fiber; **b** – across the fiber

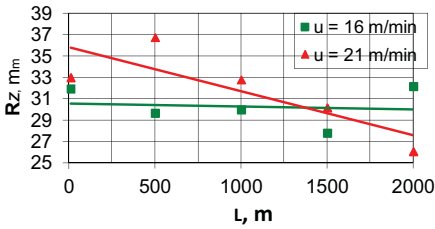
While analyzing the diagrams of results (Fig. 3a and b), when the cutting  $v$  and feed  $u$  speeds are changed, it was determined that at  $v = 54.4$  m/s and  $u = 16$  m/min the quality of processed surface is not constant when measurements are made every 500 m of cutting path  $L$ . It was determined that the  $R_z$  values had increased by average by 8 % or decreased by 5 %.

When the feed speed is  $u = 21$  m/min and cutting speed is  $v = 62.8$  m/s, the smallest surface roughness is received at the limit of 2000 m of cutting path  $L$ . This regularity does not change when the surface roughness  $R_z$  is measured across and along the wood fiber.

While analyzing the diagrams of results (Fig. 4a and b), when the feed speed is the same ( $u = 21$  m/min), and the cutting speed differs, it was noticed that the values of surface roughness  $R_z$  differ very little. The smallest value of surface roughness  $R_z$  is received at the limit of 2000 m of cutting path  $L$ . This regularity does not change when the surface roughness  $R_z$  is measured across and along the grain.

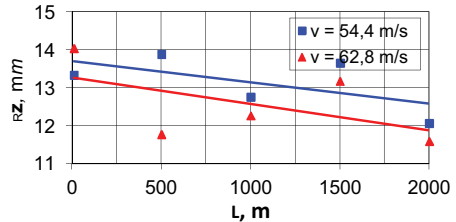


a

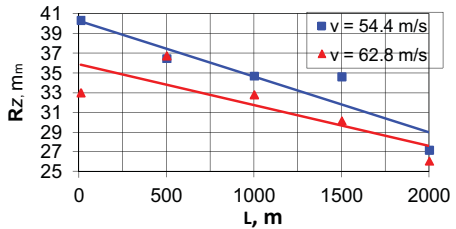


b

**Fig. 3** Surface roughness  $R_z$  of birch wood (when  $v = 62.8$  m/s):  
**a** – along the fiber; **b** – across the fiber



a



b

**Fig. 4** Impact of cutting speed  $v$  on the surface roughness  $R_z$  of birch wood:  
**a** – along the fiber; **b** – across the fiber

Initial testing of Marathon coated HSS 18 knives in pine and birch milling showed reasonable discrepancies comparing with the uncoated HSS knives performance. Main difference applies irregular and saltatory surface roughness changing in the range of 1500m long effective cutting path.

## CONCLUSIONS

1. When the cutting speed  $v$  increases, the quality of the birch surface improves. The process of shave's formation quickens and the wood fiber is cut regularly, and not destroyed by compression.
2. In the same conditions of milling regime, the quality of pine wood surface is worse than that of birch. The surface roughness of birch wood along the fiber is smaller by 4 % on average, and across the fiber – by 8 %.
3. As the cutting speed gets faster and the feed speed stays the same, the surface roughness of birch wood across and along the fiber differs very little.
4. In general researches done with Marathon coated knives confirm traditional theoretical statements about higher density wood better workability and surface quality at the same milling conditions. Still reasonable discrepancies mainly characterized by irregular and saltatory surface roughness changing comparing with the uncoated HSS knives performance have been received.



## ACKNOWLEDGEMENT

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# INTRA-LOG SAWN TIMBER QUALITY VARIATION IN NORWAY SPRUCE

Gjerdrum, P.<sup>1</sup>

## ABSTRACT

Sawmillers want to have predictable and homogeneous timber outcome from their industry. This paper analyses and discusses how well timber quality of boards from the same log, and in particular pairs of sister boards, correspond to each other, i.e. the limit for quality homogeneity. Observations from three different sawn timber outcome experiments were analysed. Varying quality traits were observed: appearance and machine strength grading, knot size and number, and board value. The results showed that 80% and 53% respectively, for two different samples, of the boards had different quality grade from their sister boards, and less homogeneity when more boards are sawn from the same log. The correlation between observations made in sister boards were modestly 0.42 for biggest green knot diameter, and almost absent for dry knots and other traits. Board value and machine strength are better correlated, 0.76 and 0.69, respectively. Relative pairwise difference for these parameters was found to follow the exponential distribution with mean 0.169 and 0.068, respectively. The results clearly indicate that there is no easy way achieving a consistent and pre-requested quality, at the same time avoiding an inherent portion of diverted quality boards.

Key words: Main yield, machine grading, pair-wise quality agreement, quality distribution, visual grading

## INTRODUCTION

Predicting sawn timber quality always was the vain hope for every sawmiller, so he could choose the right logs to cut for his own and his customers' profit. Numerous approaches have been taken in search of a fair formula; only a few recent examples will be given here to illustrate the variation in those approaches. First, one should recognise the global aspect, illustrated by the IUFRO working group 5.01.04 Wood quality modelling. One tactic would be to consider timber as the outcome of trees growing at a given site, affected by the site characteristics, which are to be passed on along with the timber. This was the idea for the European research project Indisputable Key (2009). Wood properties would then be modelled, either to predict outcome of

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timber lots (e.g. Wilhelmsson *et al.* 2002) or to model single tree quality (Moberg and Nordmark 2006). Log tomography and X-ray scanning has been another approach. Commercially available technology (Microtec 2012, RemaControl 2012) are used to predict timber quality, and a future prospect might be to increase the efficiency so as to obtain an automated optimisation of the sawmill breakdown process (Grönlund *et al.* 2005).

Nylinder (1990) introduced an industrial application using the statistical correlation between log external geometry, as can be observed in traditional log scanners, and timber quality. This idea has later been adapted at various Scandinavian sawmills (Hamar 2012, Gjerdrum 2012). Actual sawmills typically primary breakdown according to customers' specification for dimension and quality. To improve the outcome, logs with appropriate diameter are pre-sorted in two quality classes, say upper and lower. When sawing pre-sorted logs, the fraction of acceptable timber quality typically increases by some 15 per-cent-units, say from 60% to 75%. However, it has proved hard to find pre-sorting algorithms to reach beyond some 70 - 80% accept rate (Hamar 2012). One reason for this is the quality variation within any log. When sawing timber, all boards obtained from one log rarely are of identical quality. This obviously designates a *de facto* upper limit for quality consistency, whatever perfect quality prediction model for logs prior to breakdown. The objective of this paper has been to analyse and describe timber quality variation between boards sawn from the same sawlog.

## MATERIAL AND METHODS

The author has been involved in various timber quality outcome experiments at Norwegian sawmills the last decades. Logs were sawn according to the Nordic sawing pattern: Main yield in the centre, any curvature facing upwards into two (2 XLog) or four (4 XLog) boards, keeping track of log and board location in the log. Boards are always 'sister boards', *i.e.* pairwise symmetrical around the pith. Observations from three of these tests have been made available for the purpose of this paper, see overview given in Tab. 1. Sample names are codes for year of observation and geographic locality: Flesberg and Ringerike are both situated in the south-central forested area of Norway. These experiments all aimed at establishing simple and distinct algorithms for timber quality prediction, hence the small number of logs in each sample.

**Tab. 1** Overview of sawlog lots and observed traits

Sample	Top diam. cm min-max	No. of logs	Sawing pattern	No. of grades	Visual grading	Other trait info	Machine strength grading
Ring1986	20-25	88	2x	6	General classes: II' ... reject	Main reason for downgrade	-
	22-26	60	4x				
Fles2006	14-26	98	2x	-	BVI - board value indicator, NOK	Max green and dry knot diameter, mm	-
	22-31	120	4x				
Fles2010	17-22	56	2x	5	For end use: Appearance ... reject	-	SIP - strength indicating parameter
	25-34	59	4x				

## **Visual grading**

Being of quite varying origin, the observed data are not consistent. Visual grading was always done by trained operators at the sawmill in question, either in the former Nordic grades II° through VI° plus reject, *i.e.* 6 grades, for the Ring1986 sample, or according to final use: Indoor appearance, outdoor cladding, construction, pallet and reject, *i.e.* 5 grades, for the Fles2010 sample. Grading being at ordinal scale, consecutive category values (1 through 6 and 5, respectively) were assigned, and log mean grade (for 2 XLog and 4 XLog, respectively) calculated. Quality variation was calculated as the sum of absolute board grade deviation from the mean.

For the Fles2006 sample, quality assessment was combined with board output volume and actual timber sales prices at time of observation to a value indicator (BVI) for each centre board, and the value compared for symmetrical pairs of boards from the same log. In this, the relative BVI difference was calculated and analysed: the ratio of the difference in BVI between the most and least valuable boards to the pair mean BVI.

The diameter of the biggest green knot and the number of knots over 5 mm were observed in the Fles2006 sample. Similar observations were done for dry knot, for resin pockets and for other downgrade traits. To reduce all observations to a scalar value, a joint trait index (JTI) was calculated by adding these figures. Several JTI algorithms were tested, but, as will be shown, none very successful.

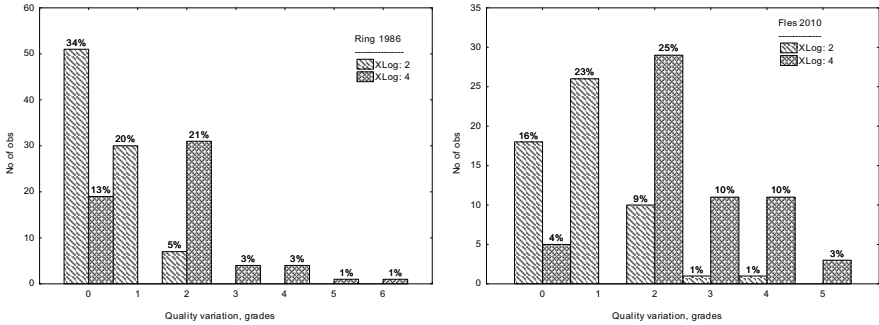
## **Machine strength grading**

One set of boards, Nume 2012, was machine strength graded applying the Dynagrade equipment for industrial application. For each board the strength indicating parameter (SIP) was recorded, and the observations compared for symmetrical pair of boards from the same log. SIP was treated much the same as BVI, thus the pair mean SIP and the ratio of difference in SIP between the strongest and the weakest board to this mean were calculated.

# **RESULTS AND DISCUSSION**

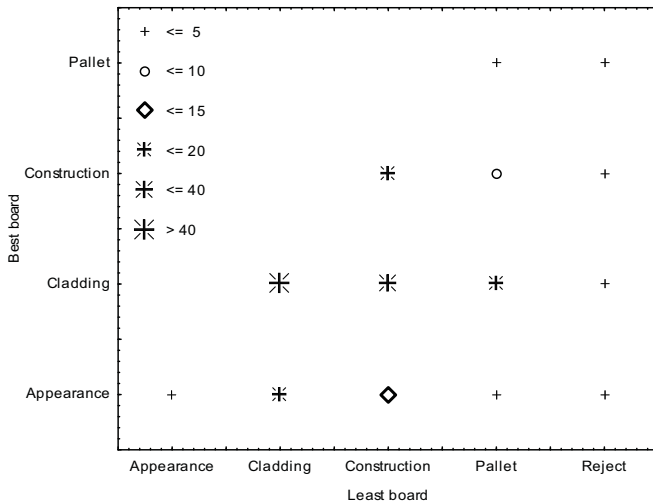
## **Quality grading**

Quality variation, *i.e.* sum of absolute deviation from mean grade for all boards from a log, is illustrated in Fig. 1. The overall impression is that of substantial quality variation within logs, and, as can be expected, more variation with more boards in the centre yield. The disparity between the two samples probably is due to distinctly different grading rules in combination with random variation. Most homogeneous grade was found for the 2 X log graded according to the traditional rules in Ring1986; here two thirds of the board pairs were of the same grade - usually grade V. In 4 X Log graded for final use in Fles2010 only four of fifty logs had similar grade for all boards.



**Fig. 1** Histogram for quality grade variation within logs for Ring1986 (left) and Fles2010 (right) samples

The problem is further illustrated for the Fles2010 sample in Fig. 2. Although most boards hold the cladding or construction quality, for any grade of the best board in a log, also all lower grades were found in other boards from the same log in this sample.



**Fig. 2** Frequency plot for least vs. best board quality for the Fles2010 sample

### Pairwise correspondence for sister boards

The correlation was modest  $r = 0.42$  for biggest green knot diameter, and almost absent for dry knots and the joint trait index (Tab. 2). These traits all had a dual distribution, *i.e.* a certain fraction of the boards, those listed in Tab. 2 under heading 'zero', had no observable object for this trait. The rest of the observations had a skewed distribution with a tail to the right, looking like the lognormal distribution.

All board pairs had almost the same volume, only with small allowance for clear-cutting, so most of the BVI variation within pairs was mainly due to quality related timber price variation. The correlation was 0.76.

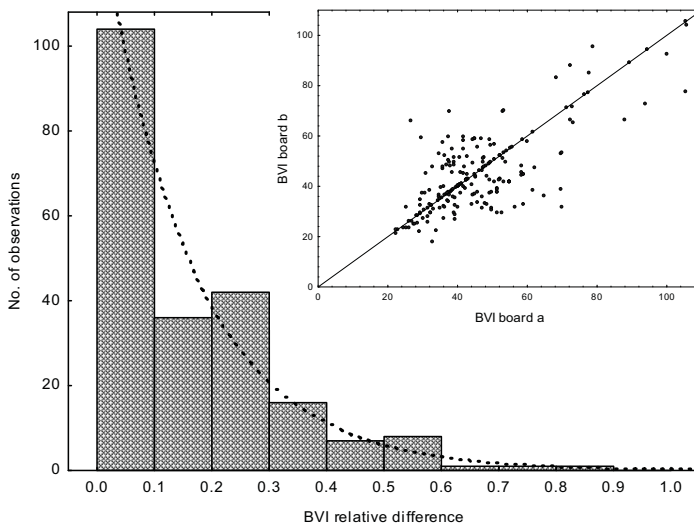
The correlation for machine strength grading was 0.69 (Tab. 2). Industrial machine strength grading is, however, applied with a certain accuracy tolerance. Consequently, a part of the missing correspondence might be due to this industrial noise.

**Tab. 2** Statistical information and correlation between observations in pairwise symmetrical boards for important traits.

Trait	Mean	Std.dev.	Distribution		Correlation for board pairs, $r_{\text{pair}}$	
			zero	rest		
Biggest green knot	mm	19.9	10.7	dual 14 %	0.42	
Biggest dry knot	mm	4.3	7.2	dual 71 %	skewed, tail to the right	0.08
Joint trait index <sup>a)</sup>	JTI	50.3	34.1	dual 4 %		0.13
Board value indicator	BVI	45.1	15.7	- 0 %		0.76
Machine strength indicator	SIP	$6.6 \cdot 10^6$	$0.62 \cdot 10^6$	- 0 %	normal	0.69

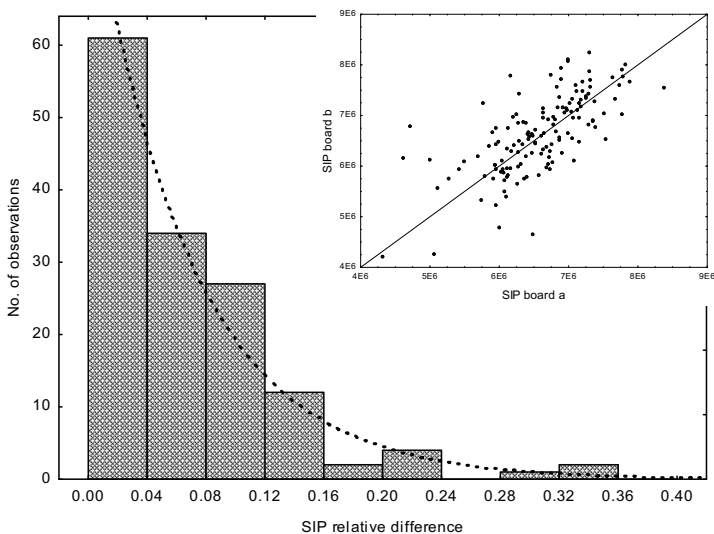
<sup>a)</sup> Sum of (biggest knot + number of knots) for green and dry (double weight) knots and other traits

The BVI relative difference, *i.e.* the BVI difference between pairs divided by pair mean BVI, is extremely skew distributed, Fig. 3. It matches fairly well the exponential distribution with mean 0.160 and standard deviation 0.169. The observed range for BVI relative difference is from 0.0 to 0.85; theoretical difference is from zero when both boards have identical value, to two, when one board has no value at all.



**Fig. 3** Distribution for relative difference in board value indicator BVI for sister boards. The exponential distribution function is shown in dotted line.

Upper right corner: Bivariate correspondence in BVI for sister boards.



**Fig. 4** Distribution for relative difference in machine strength SIP for sister boards.

The exponential distribution function is shown in dotted line.

Upper right corner: Bivariate correspondence in SIP for sister boards.

SIP relative difference was extremely skew distributed, Fig. 4, similar to its analogue companion relative BVI difference. It matches fairly well the exponential distribution with mean 0.068 and standard deviation 0.063. The observed range was from 0.0 to 0.36. The exponential distribution requires the mean and the standard deviation to be of the same magnitude, which is reasonably well met, both for relative BVI and relative SIP difference.

## CONCLUSIONS

The results clearly indicate that there is no easy way achieving a timber lot of consistent and pre-requested quality, at the same time avoiding an inherent portion of diverted quality boards. This should be of great importance for sawmill operation. However, illustrating the challenging nature of timber quality prediction, the results should be verified in a wider range of situations: log supply, sawmill set-up, customer requirement, etc., before decisive conclusions can be drawn.

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# THE BASIC INFLUENCING FACTORS ANALYSIS OF THE LATVIAN WOOD PROCESSING INDUSTRY

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## ABSTRACT

The global wood and timber market was developing very rapidly in the last pre-crisis decade, which contributed to the demand and the global trade growth in this sector, but today this market are also able to slowly develop. With forests covering more than 50% of its territory (1.6 times the world average), Latvia is one of the most forested EU member states. Over the last 70 years, the forested area has nearly doubled while standing volume has increased 3.6 times, reaching 631 million cubic metres. About 50% of forests are owned by the state. The forest sector's share of Latvia's gross domestic product is about 5%; in 2010, the value of products turned out by the sector reached LVL 1.5 billion (EUR 2.1 billion). Primary wood processing is generally based on modern technologies meeting the requirements of the global market both in terms of productivity and product quality. As a result, primary wood processing has built a stable foundation both for exports and the development of further processing and the production of higher value added products.

In the study, the influencing factor selection with sociological research method (analysis), factor analysis and expert's method was used. From the study results it can be concluded that the key affecting factors whether to influence Latvia's wood processing development are the following: a roundwood and other materials available for the production, a cost and pricing imbalance of products, a trends in the sales markets and competitor countries and a new development direction of wood products and other. It should be noted that each of the significance of this factor may vary considerably shorter period of time, i.e. year. The main conclusion is that the Latvian wood processing industry to significantly affect the ability to sell the primary products to local needs, as well as their ability to sell their products with general trends in the global wood products markets are mutually uncorrelated, because they are to small players in this market.

**Key words:** primary wood processing sector, influencing factors, Latvia

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## INTRODUCTION

Despite the global economic and financial crisis, most of the Latvian forest sector enterprises successfully overcome. Latvian forest sector value added in the last ten years increased significantly from 208 million lats in 2000 to 601 million. lats in 2010. Latvian forest sector in the economy over the last decade ranged from 3.5 to 5.5% range and in 2010 the share of GDP was 4.7%.

During the period from 2000 to 2010, the Latvian forest sector net turnover has tripled, reaching 1.5 billion lats, which accounted for the highest turnover of wood and wood products (NACE 16.group), i.e. ~ 67%, leaving behind furniture (NACE 31.group) with 26% and forestry and logging (NACE 02.group) to 7%.

World economic and financial crisis is only a short period of time been able to influence the Latvian forest sector export development as early as 2010, their volume, compared with the previous year, increased by about 45% to almost 1,02 billion lats in export value, which is the second largest increase in value over the period 1993 to 2010. A Latvian forest industry export in 2010 was 22% of total national exports. All the time the largest export volume was achieved in 2007, the Latvian foreign markets for manufactured products reached nearly 1.06 billion lats. Also in 2010 the Latvian forest industry export value reaches 1 billion lats border and just slightly from 2007, which was reached before the maximum value of exports. Latvian forest sector and will have wood products (marketable new products) export potential. Latvian forest industry structure shows that currently the largest share of primary products as sawn timber, lumber and energy wood export then ranks panels and further wood products export.

Latvian forest sector can be divided into three sub-sectors, i.e. primary sector, further processing sector and furniture. Each of the sub-sector is produced according to the product and they are:

- primary wood processing sector produced - sleepers, sawn softwood, hardwood sawn timber, pulp chips or chips, fuel chips or shavings, wood in the rough, treated railroad ties and wood chips;
- further processing of wood products sector produced - pellets, briquettes, mulch and the like, other chips products, coniferous sawnwood, hardwood sawnwood, plywood, boards, sheets, pads, pallet collars, boards, barrels, chests, boxes, drums;
- furniture manufactured wood products sector - wood doors, frames, jambs and thresholds, wooden windows and French windows, balcony glass doors, joinery products, Prefabricated wooden buildings, wooden stairs, ladders, and construction, decorative wood products, sofas and chairs , other wood products, furniture components, office furniture, kitchen furniture, bedroom furniture and other furniture.

## MATERIALS AND METHODS

According to the research objectives and tasks to obtain the results was based on such scientific research methods - sociological research method (analysis), analysis of the factors and expert method.

As one of the main research methods that work - analysis of documents (Sociological..., 1981) belonging to the sociological research methods (Research..., 2005). In order to study the people (public) behavior of various economic activities, depending on the study task, can be used both quantitative and qualitative research methods. A quantitative study is a social processes and social phenomena through empirical study of social research methods. It explores certain quantitative characteristics of the social action of certain objects or subjects. Factor analysis is a statistical method that not only allows you to find the factors in a number of variables based on commitment, but also to evaluate the closeness of the obligations between the factor and the observed features, namely, to answer the question, how big a factor is the proportion of each feature (Factor..., 2012) The expert method in the forest sector and related sectors are often used in the current trend of understanding and appreciation can only professionals. Therefore, to select the key competitiveness factors were used precisely this method using 5-point system so giving a significance level of each factor (Tunkele..., 2010).

## RESULTS AND DISCUSSION

According to the experts carrying out the methods were identified 6 key factors influencing the primary wood processing sector and they are – the roundwood and other raw materials availability, raw materials and other costs of compliance with the market price, private forest owners' activity, wood products in development areas, development trends in sales markets and development trends in the countries (Fig. 1.).

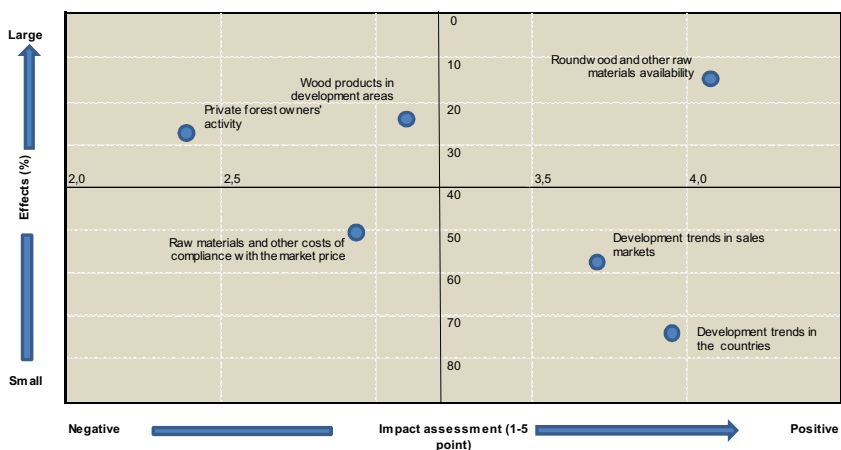


Fig. 1. Latvian wood-processing sector, the influence of the size and assessment

It should be noted that each share of the significance of this factor may vary considerably shorter period of time (e.g., year). When analyzing the Latvian primary wood processing sector of the market norm, the author would like to draw attention to some of them specific to Latvia, that is distinguished wood utilization trends in the country of the foreign trade markets, the domestic market sector enterprises mainly takes the form of their product sales to intermediaries who export or any other businesses that add value to it, and exported, as well as a significant domestic market exists only in the energy of wood products, joinery and furniture.

### **Roundwood and other raw materials available for wood processing**

The main factor influencing the primary wood processing sector is the development of round timber and other raw materials available for processing. According the experts, Latvian forest industry is directly or indirectly dependent on the availability of roundwood. Currently, only 16 % of wood products that can be economically feasible to manufacture, transport within a reasonable distance away from harvesting or primary wood processing company. Because ~ 47% of primary sector and ~37% further processing sector is able to indirectly affect the place of logging and primary wood processing enterprises.

### **Raw materials and other costs compliance with the market price**

Latvian forests industry primary wood processing sector to affect the raw material price, their fluctuations and trends. Latvian forests sector total production volumes are dependent on the availability of wood processing. If the market will decrease the demand for timber, it will upset the market balance between supply and demand, change the product prices. Lumber production cost structure of the main item of expenditure is the cost of sawn timber (Latvian, ranging from 60 - 70% of the total cost), resulting in lumber prices fall event is the offset of the purchase price of sawlogs cut.

### **Private forest owners' activities**

The wood raw material market is a key factor in the private forest owners' activities, because there is some interaction between the wood raw material prices and harvest volumes are affected as a result of the total input flow of sawlogs and veneer to the processing sectors, from time to time affecting the total production in the Latvian primary processing enterprises. But not always the amount of timber resources from forest owners' dependency of sawn timber price levels if harvesting costs are lower than the price of sawlogs, the private forest owner's decision to carry out logging affects the expectations of price changes and short-term price forecasts. Compared to the 2008th and 2010 the primary sector, the main product as sawlogs, veneer logs and pulpwood prices, there are no significant differences in range of prices (Table 1), but important difference is that 2008 prices are significantly lower compared with 2007, while, 2010, private forest owners are aware of the situation that exists in the market

for low round assortment of prices and sales of major problems in pulpwood production.

**Table 1** The roundwood assortment of price comparison between 2008 and 2010 in Latvia (lats)

Assortment	2008	2010	Assortment	2008	2010
Spruce sawlogs (10-13,9 cm)	31	29	Aspen sawlogs (18 cm)	24	23
Spruce sawlogs (10-18,9 cm)	37	34	Aspen sawlogs (22 cm)	33	26
Spruce sawlogs (19-27,9 cm)	43	39	Aspen sawlogs (A class)	40	32
Spruce sawlogs (28+)	44	41	Pine pulpwood	26	28
Pine sawlogs (10-13,9 cm)	33	29	Spruce pulpwood	26	28
Pine sawlogs (10-18,9 cm)	39	32	Birch pulpwood	25	28
Pine sawlogs (19-27,9 cm)	44	37	Aspen pulpwood	21	17
Pine sawlogs (28+)	45	38	Veneer (B class)	54	33

Private forest owners' decision to conduct logging also affects the rest of the assortment and price changes. According to experts the information collected is expected to range the wood price in the short term prices will rise, while pulpwood prices will fall. The range of price fluctuations will lead to reduced supply from private forest owners. However, this process has a positive effect in the above ranges are increasingly being used in the energy market fuel wood production. Energy market processes are more stable and predictable, which will also be beneficial in bulk and particle board production.

### **Wood products in development areas**

According to experts, traditional wood products manufacturing trends Latvian practice development opportunities have been exhausted and further development of multi-fuel wood production and species such as aspen and gray alder processing. As a result, the main potential lies in the distant pulpwood and lumber processing. If the next few years pulpwood prices will fall and will hold steady or such longer period will not exceed 25 lats per cubic meter, then continue in pellet production capacity of Latvian. In turn, joint expert information provided by the timber recycling higher value added products will not be active, because there is no projected increase in purchasing power in domestic market, new construction industry, the rapid growth of the local construction industry, changes in state and local government procurement in favor of wood use.

### **Development trends in the sales markets and competitor countries**

The main market for the primary wood processing sector is the construction market. The author argues that a nation's primary forest sector growth is dependent on processes of global and local construction markets. Analysis of secondary sources of information and data for a survey of industry experts is expected to stagnation or low growth rates in all construction markets such as Europe, USA and Japan, except for China and the Middle East and North Africa markets. This is evidenced by the construction of Europe's leading business research group "Euroconstruct" releases and the prospects that the 2020 construction rates in Western Europe will grow less than 2% per year, while Eastern European countries of 4.5 - 5% per year. By contrast, in rich Western countries, the construction pace will be slow, i.e. ~ 1.5% per year, mostly

in the renovation and construction of new housing bill, but the pace of construction in southern European countries in 2020 will be only slightly above the 2010 level.

After the above information, it can be argued that as a lumber product markets are expected in a rapid change, however, can not believe that the market equilibrium will not be disturbed. Already, all the lumber-producing potential capacity exceeds the volume of production. Latvian forest industry primary wood processing sector should take into account the primary factor in the sales markets are characterized by instability, because of the total lumber production of hasty growth from changes in market equilibrium, which will lead to price fluctuations. Looking at some major Latvian forest industry primary wood processing sector produced timber use in construction, it is concluded that there is a correlation between them.

### **Development trends in the competitor countries**

To summarize secondary information sources and industry experts, it can be concluded that the U.S. construction market is expected to slow after-care to note that the current volume of construction in the country is historically low. By contrast, the Japanese construction market stabilizes short-term disaster and long term are not expected sharp increase in the pace of construction. At the beginning of 2016 the Japanese construction market is forecast to fall 10% of the nation's rapidly aging and other negative demographic characteristics. In turn, the Chinese construction market is not available for objective long-term forecasts, but estimates suggest that China is currently the second largest softwood lumber in the world consumer member and an annual growth of imports in the coming years is projected 15 - 20% boundary. According to experts of the information provided, that the present lack of objective information, it is difficult to predict trends in the construction market in the Middle East and North Africa region, but markets have a negative impact on political instability, but positive - demographic characteristics.

Although it is believed that the demand for certain timber products in global markets affect the overall production, but can not unequivocally state that it will directly affect the Latvian forest industry primary wood processing sector production levels. In 2010, the world's largest softwood lumber producer share are USA (23.0%), Canada (20.6%), Germany (11.6%), Russia (10.2%), Sweden (9.3%), Austria (5.2%), Finland (5.1%), France (3.8%), Australia (2.3%), Britain (1.7%), Romania (1.5%), Latvia (1.4%), Norway (1, 1%), Spain (0.8%), Czech Republic (0.8%), Belgium (0.7%), Italy (0.4%), Denmark (0.3%) and Netherlands (0.1% ). When analyzing the data suggests that some correlation exists between the Latvian softwood lumber exports to certain countries' markets, but it does not mean that in certain countries or groups (Japan, USA and Europe) of consumption is a key factor in determining trends in the volume of Latvian exports.

## CONCLUSIONS

The six 6 key factors influencing the Latvian forest industry primary wood processing sector are the roundwood and other raw materials availability, raw materials and other costs of compliance with the market price, private forest owners' activity, wood products in development areas, development trends in sales markets and development trends in the countries. The main factor influencing the Latvian primary wood processing sector is the development of round timber and other raw materials available for processing and only 16 % of wood products that can be economically feasible to manufacture, transport within a reasonable distance away from harvesting or primary wood processing company. Lumber production cost structure of the main item of expenditure is the cost of sawn timber, resulting in lumber prices fall event is the offset of the purchase price of sawlogs cut. The private forest owners' activities are an important for Latvian primary sector, because there is some interaction between the wood raw material prices and harvest volumes are affected as a result of the total input flow of sawlogs and veneer to the processing sectors. Traditional wood products manufacturing trends Latvian practice development opportunities have been exhausted and further development of wood production Looking at some major Latvian forest industry primary wood processing sector produced timber use in construction, it is concluded that there is a correlation between them. Latvian forest industry primary wood processing sector development is important to realize that this sector will be unable to regulate the growth of domestic demand, resulting in no significant impact on the sector's sales of products manufactured for domestic consumption. Latvian primary wood processing sector producer's markets share in the world is too small, so the trend of sales markets and competitors countries will not affect manufacturers' ability to promote product sales.

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# GLUABILITY AND BONDING QUALITY OF CELLULAR WOOD MATERIAL

Iejavs, J.<sup>1</sup> & Jakovlevs, V.<sup>2</sup>

## ABSTRACT

Invention of light weight panel with trade mark of Dendrolight is one of the most distinguished wood industry innovations of last decade. At present three layers cellular wood panels have wide non structural application in furniture and door industry. For further product development study on cellular wood material gluability with other wood based materials was necessary. The aim of the research was to evaluate the gluability and bonding quality of pine (*Pinus Sylvestris* L.) cellular wood material with solid wood (pine, ash and thermally and mechanically treated grey alder) and wood based panels (oriented strand board, high density fibreboard and birch plywood). Thermoplastic polyvinyl acetate adhesives are typical for three layer cellular panel gluing. To increase the thermal resistance of the products emulsion polymer isocyanate Kleiberit EPI 304.4 and polyurethane Kleiberit PUR 501 adhesives were evaluated as binders for three layer cellular wood panel production. There were 15 specimens manufactured of each of twelve cellular wood material, adhesives and top layer materials combinations. To evaluate bonding quality of panels tensile strength perpendicular to the plane of the panels was evaluated according to standard LVS EN 319:2000. Fracture mode of the glued joint as extra parameter was evaluated. The initial research shows that emulsion polymer isocyanate and polyurethane adhesives with good results can be used for three layer cellular wood material production for both solid timber and wood based panels. The average tensile strength perpendicular to the plane of the panels varies from 0.31 N/mm<sup>2</sup> (panels with oriented strand board top layers) to 1 N/mm<sup>2</sup> (panels with pine or plywood top layers). Top layer significantly influences the fracture mode of the three layer cellular wood panels.

Key words: dendrolight; sandwich panel; gluability

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## INTRODUCTION

Three layer cellular wood panels consists of core layer made of solid wood with grooves and two top layers made of thin solid wood or wood based panels. Since 2010 when industrial production of panels was started up to now three layer cellular panels has wide non structural application in furniture and door industry. Initial research on cellular wood material made of aspen (*Populus tremula* L.) wood shows that tension strength perpendicular to the plane of three layer cellular wood material panels varies from 0.46 to 0.86 MPa (Iejavs at al., 2007). Thermoplastic polyvinyl acetate adhesives are typical for three layer cellular panel gluing. To increase the thermal resistance of the products made of pine (*Pinus sylvestris* L.) cellular wood material emulsion polymer isocyanate Kleiberit EPI 304.4 and polyurethane Kleiberit PUR 501 adhesives were evaluated as binders for three layer cellular wood panel production. There were 15 specimens manufactured of each of twelve cellular wood material, adhesives and top layer materials combinations. To evaluate bonding quality of panels tensile strength perpendicular to the plane of the panels was evaluated according to standard EN 319:1997. Fracture mode of the glued joint as extra parameter was evaluated. The essential goal of the research was to evaluate the bonding quality between pine cellular wood material and solid timber (pine, ash and thermo mechanically treated grey alder) and conventional wood based panels (high density fibre board, birch plywood and oriented strand board) glued with adhesives with higher thermal resistance instead of thermo plastic polyvinyl acetate adhesive.

## MATERIAL AND METHODS

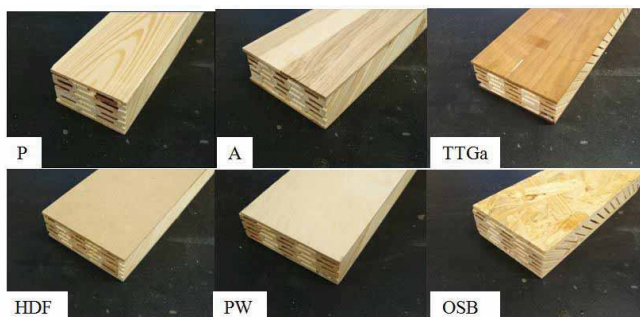
### **Manufacture of the Scots pine cellular material**

As a raw material for cellular wood material production pine (*Pinus sylvestris* L.) timber was used with nominal dimensions: thickness – 32 mm, width – 112 mm and length – 4200 mm. Cellular material was manufactured industrially on the automatic production line of the company Dendrolight Latvija Ltd. All significant wood defects were removed before timber finger jointing. Technical data of the finger jointed pine wood: finger length - 10 mm, finger pitch - 4 mm, tip gap - 1 mm. Finger joint end pressure 12 MPa was applied at least for five seconds. The average moisture content of the boards was 12%. One component polyvinylacetate (PVA) adhesive Cascol 3353 was used for face gluing of the boards. According to the standard EN 204:2001, the moisture resistance class of adhesive Cascol 3353 is D3. After finger jointing fingers are visible on the flat face of the timber. During manufacturing process and before testing all materials were kept in constant atmosphere at  $20\pm 2$  °C temperature and relative humidity of  $65\pm 5\%$  to prevent wood material moisture changes. The thickness 28 mm and width 106 mm were obtained after four side planing operation. After the planing operation all boards were cut to 2010 mm length. After that 8 double faced grooves were cut into longitudinal direction in the flat faces of boards with the following dimensions of the grooves: depth of 24 mm, pitch of 6.4 mm and width of

3.2 mm. Four layers of grooved boards were used to produce cellular wood material blocks. Each layer was aligned horizontally in 90 degree direction against the previous layer. Cellular material blocks were produced with continuous operation hot press. Oscillation method was used to ensure glue spread from 200 to 300 g·m<sup>-2</sup> between block layers. Pressing was carried out with pressure 0.2 MPa at 60 - 75 °C temperature and pressing time was 6 min. After pressing pine cellular wood material blocks with dimensions: thickness 112 mm, width 1350 mm and length 2500 mm were obtained.

### Manufacture of the three layer cellular wood material panels

In total twelve types of cellular wood material panels were manufactured, six for each adhesive. 42 mm thick cellular wood material slices with length and with 300 mm were used as a core material for three layer cellular wood material production. The average density of cellular wood material was 300 kg·m<sup>-3</sup>. Polymer isocyanate Kleiberit EPI 304.4 with hardener Kleiberit 808.0 (EPI) and polyurethane Kleiberit PUR 501 (PUR) adhesives were evaluated as binders for three layer cellular wood panel production. Pine (*Pinus sylvestris* L.), ash (*Fraxinus* L.) and thermo mechanically treated grey alder (*Alnus incana* Moench) solid timber and wood based panels: high density fibre board; birch plywood and oriented strand board were used as top layers for three layer cellular wood panel production. The thickness of top layers in all types was 4 mm except oriented strand board where 8 mm thick panel was used. The density of top layers were: pine 470 kg·m<sup>-3</sup>; ash 670 kg·m<sup>-3</sup>; thermo mechanically treated grey alder 720 kg·m<sup>-3</sup>; high density fibre board 800 kg·m<sup>-3</sup>; birch plywood 700 kg·m<sup>-3</sup> and oriented strand board 620 kg·m<sup>-3</sup>. Photos of three layer cellular wood material panels with six different top layers are presented in Fig. 2. Both adhesives in the top layer gluing operations were applied manually with a hand roller, and the average glue spread measured by weighing method was from 150 g·mm<sup>-2</sup> for EPI adhesive to 170 g·mm<sup>-2</sup> for PUR adhesive. The cold setting hydraulic press was used in panel manufacturing with plane pressure 0.2 MPa and pressing time of 1 hour. The thickness of the three layer cellular wood material panels covered with oriented strand board was 58 mm, in other types 50 mm.

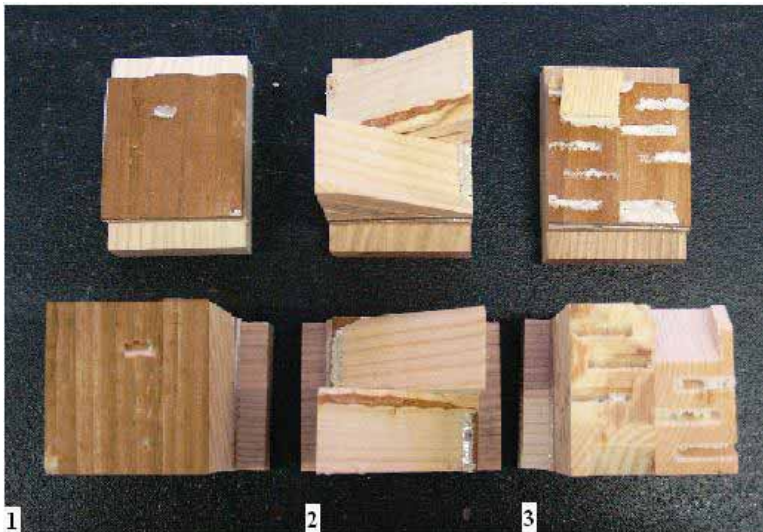


**Fig. 1.** Illustration of the panels with six different top layers: P – solid pine; A – solid ash; TTGa – thermo mechanically treated grey alder; HDF – high density fibre board; PW - birch plywood and OSB - oriented strand board.

All specimens were marked with adhesive label EPI or PUR, with top layer label according to Fig. 1 and specimen No. from 1 to 15 for each panel type.

### Test methods and data processing

Before testing all specimens were conditioned in the standard atmosphere ( $20\pm 2$  °C;  $65\pm 5$  %) to reach the constant mass. 15 specimens of each of twelve panel types were used to determine tensile strength perpendicular to the plain of the panel according to standard EN 319:1993 and technical specification project prCEN/TS 00112189:2010. Tensile tests were carried out in Forest and Wood Product Research and Development Institute on the ZWICK Z 100/TL 3S material testing device. All specimens were stressed until rupture. After tensile test typical type of fracture of the specimens were evaluated for each panel type according to Fig. 2.

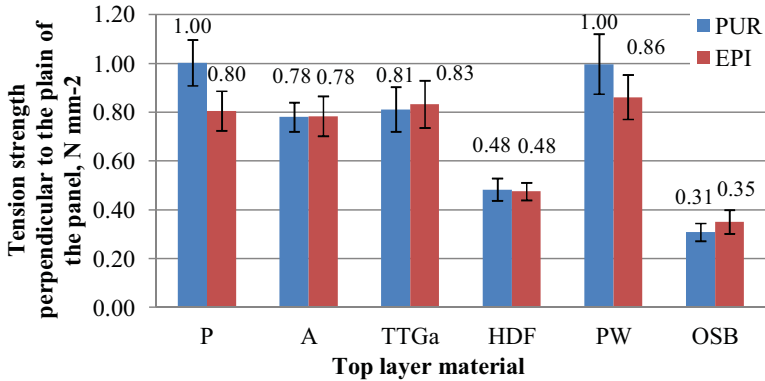


**Fig. 2.** Typical types of fracture: 1 – in top layer;  
2 – in core layer and 3 – in glue line.

In order to compare the mean values of twelve types of panels tensile strength perpendicular to the plane of the panel acquired from at least 11 specimens, independent sample t-test with p-value method ( $\alpha = 0.05$ ) was used. If t-test's p-value is lower than 0.05 the mean values between panels differs significantly, but if p-value is higher than 0.05 the mean values between panels did not differ significantly. Mean values and 95% confidence interval for mean presented in Fig. 3. Typical types of fracture for each of twelve types of panels presented in Fig. 4. When optimal gluing parameters are used fracture occurs in top or core layer, when gluing parameters are not optimal fracture occurs in glue line.

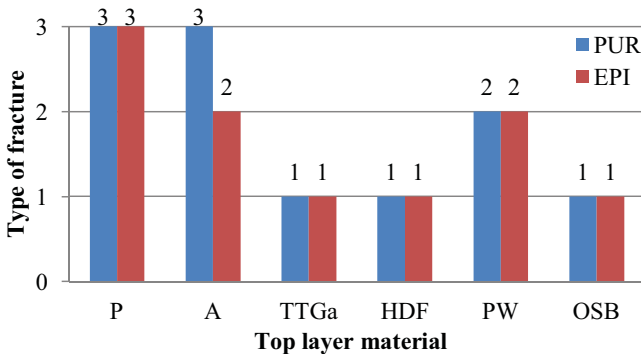
## RESULTS AND DISCUSSION

In total 158 specimens were tested for 12 panel types to evaluate the tensile strength perpendicular to the plane of the panels and type of fracture. Tensile strength mean values and 95% confidence interval for mean for each panel top layer and adhesive combination presented in Fig. 3.



**Fig. 3.** Tensile strength perpendicular to the plain of three layer cellular wood material panel in dependence of top layer material: P – solid pine; A – solid ash; TTGa - thermo mechanically treated grey alder; HDF – high density fibre board; PW - birch plywood; OSB - oriented strand board and adhesive: PUR - polyurethane Kleiberit PUR 501 and EPI - emulsion polymer isocyanate Kleiberit EPI 304.4.

The highest tensile strength or better bonding quality was observed when cellular wood material was covered with pine or plywood and polyurethane adhesive Kleiberit PUR 501 was used as binder. The obtained average tensile strength in both cases was 1.0 MPa and significant difference between them was not observed. Fracture occurred in glue line for panels with pine top layers and in core layer for panels with plywood top layers (Fig. 4).



**Fig. 4.** Type of fracture in dependence of top layer material and adhesive: 1 – in top layer; 2 – in core layer; 3 – in glue line.

The significant difference between panel tensile strength mean values were not observed when cellular wood material was covered with pine solid timber or plywood (with adhesive Kleiberit EPI 304.4), ash solid timber or thermo mechanically treated grey alder (with both adhesives). The average value varies from 0.78 to 0.86 MPa. The above mentioned panel types except panels with solid pine and solid ash top layers characterized with fracture in top or in core layer and it means that adhesives and gluing parameters used were optimal for three layer cellular wood material panel production. Panels glued with solid pine top layers with both adhesives and panel glued with solid ash top layers with polyurethane adhesive does not obtain optimal tensile strengths values and type of fracture. The fracture mode can be improved by finding optimal gluing parameters suitable for cellular wood material gluing with solid pine and solid ash wood. The significantly lower tensile strength values were observed: for panels with high density fibre board top layers (average values for both adhesives 0.48 MPa) and for panels covered with oriented strand board (average value 0.31 MPa for PUR adhesive and 0.35 for EPI adhesive). For all four types of panels fracture occurred in top layer and glue bond ensure higher tensile strength than internal tensile strength of boards. If we compare the influence of adhesive type on the tensile strength of the three layer cellular wood material panels we can see that in most panel types adhesive type did not influence tensile strength, with two exceptions: when panels covered with solid pine or birch plywood, in these two cases panels glued with PUR adhesive showed significantly higher tensile strength values compared with panels glued with EPI adhesives. In all cases except one the adhesive type did not influence the fracture mode of the panels, only when panels covered with solid ash types of fracture differs.

## CONCLUSIONS

The results showed that tensile strength and bonding quality perpendicular to the plain of three layer cellular wood material panels depend directly on tensile strength properties of top layer material or tensile strength properties of cellular wood material. Obtained average tensile strength properties for three layer cellular wood material panels varied from 0.31 to 1.00 MPa. Both adhesives Kleiberit PUR 501 and Kleiberit EPI 304.4 with good results can be used as binders between cellular wood material and solid wood or wood based panels with minor influence on tensile strength and type of fracture of panels.

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# GREY ALDER PRETREATMENT BY STEAM EXPLOSION FOR SELF ADHESIVE COMPOSITES

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## ABSTRACT

A batch steam explosion pretreatment (SE) was applied to grey alder (*alnus incana* (L.) Moench) chips to obtain self binding fibrous lignocelluloses' complex. SE was generated by water vapours without any chemical catalyst. The structural impact of SE on the chips investigated by such aspects as: mass loss (depending on severity of SE, moisture and chips size); changes of microstructure; components output; changes of bulk density. The goal of the study was to get information about the pretreated lignocelluloses' complex which further was used for a binder-less board production. There was investigated that mass loss of the chips mostly depends on pretreatment conditions and increases with increasing SE severity. The bulk density of the pretreated chips decreases at least by 1.5 times and it depends on the chips size and the severity of SE. The microstructure of pretreated lignocelluloses' complex is greatly modified; however it includes the same content of lignin and cellulose as in untreated wood. While at least half of the origin hemicelluloses content is lost during SE.

Key words: grey alder, steam explosion pretreatment, microstructure, component fractionation, bulk density.

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## INTRODUCTION

A wide range of commercially produced bio-based composites are bonded with synthetic adhesives matrix. These adhesives are mostly obtained from petroleum and gas products; therefore they are not environmentally friendly and have a trend to increase in costs. It is well known that bio-based materials can be bonded without synthetic adhesives. Some of the newest research in this area involves the use of enzymes, surface activation, biotechnology, chemical modification, and cold plasma (Rowell 2007).

In 1926 Mason patented wood disintegration technology generating the substances from processed material with self-adhesive properties (Mason 1926). The process factors include high temperature steam pressure and time followed by fast decompression, physically and chemically affecting all wood compounds. The steam exploded (SE) substrate further was used for commercially wet process self-sustaining fibreboards production at the Masonite Corporation (Mason 1930). There was investigated that bonding mechanism of the pressed fibreboards depends on sufficient sugar content in the substrate (Turner 2000; Rowell et al. 2000).

Today SE technology is one of leading pre-treatment methods for second generation bio ethanol and bio gas production from lignocelluloses (Miller 2007). However the SE process is still potential method investigating new possibilities to obtain bio-based composites from fast growing wood species or agricultural wastes (Velasquez et al. 2003; Mancera 2008; Shao et al. 2009). All the authors were proven that lignin is liberated from the cell wall to the fiber surface during SE and it is the most important component making self binding mechanism of further pressed composites.

In spite of many works done pretreating biomass by SE there is a lack of information about the biomass physical characteristics such as yield and bulk density. Therefore the present paper includes the mentioned details as well as microstructure changes of grey alder wood chips pretreated in SE process. The goal of the study was to get the information about grey alder lignocelluloses' complex which further was used obtaining the binder-less fibreboard (Tupciauskas et al. 2009; 2011).

## MATERIAL AND METHODS

Latvia's fast growing hardwood species of grey alder (*alnus incana* (L.) Moench) was crushed by wood chipping machine ("Bruks", Sweden), cleaned of bark and air-dried to moisture content varying from 6 % to 11 %. One part of the chips could be characterised by mesh fractionation with 20 mm holes. Other part of the chips was crushed (by "Retsch", Germany) to particles which could be characterized by mesh holes diameter of 2 mm. The particles fraction equal to and less than 0.4 mm was removed out.

The SE for above described materials was done with saturated steam in a batch reactor (Tupciauskas et al. 2009) at temperatures 200 °C and 235 °C generating vapour pressure 1.6 and 3.2 MPa respectively. The duration of the pretreatment at 200 °C was 10 min but at 235 °C – 0.5, 1, 2, and 3 minutes. Common logarithmic values of the severity factor ( $R_0 = t \cdot \exp(T-100)/14.75$ ) of the SE conditions was used to present the treatment temperature (T) and time (t) by a single variable (Heitz et al. 1991). The pretreated samples depending on the pretreatment time could be defined as SE0.5, SE1, SE2, SE3, SE10 and correspond to the severities ( $\log R_0$ ) 3.67, 3.97, 4.28, 4.45, 3.94.

The particles before and after SE treatment were observed using a Vega TS 5136 scanning electron microscope (SEM) operating at 15 – 20 kV, after sputter coating with gold. Mass loss of the pretreated materials determined by weighing the mass before and after the SE taking into account the moisture. Making the simple component fractionation of the pretreated material (all processes at room temperature), first, the soluble parts removed by adding water (1:4). Lignin is extracted from the residual by solving it in 0.4 % solution of NaOH (1:4) wherefrom it is precipitated by adding hydrochloric acid to neutralize the solution. The precipitated mass is rinsed in water to remove the remnant of sodium chloride before filtration. After drying in air the filtrate turns into powder presented as steam-exploded lignin (Gravitis et al. 2010). The bulk density of dry matter of both pretreated and unpretreated material determined in  $\text{kg m}^{-3}$  using standardized method (CEN/TS 15103 2005).

## RESULTS AND DISCUSSION

As it is known a certain part of biomass volatiles out during SE. This part is defined as mass loss and contains degraded hemicelluloses (Gravitis 1987). Mass loss of SE mostly depends on severity of the process and it indicates tight correlation shown in Fig. 1. The most unstable polymer of grey alder wood degrades irreversibly up to 20 % at  $\log R_0 = 4.45$  losing almost all its content. The mass loss variation at  $\log R_0 = 3.97$  depends on moisture content of the particles and slightly decreases with the increasing moisture content from 6 % to 11 % (Fig. 1).

While size of the particles show no significant impact on the mass loss. The samples SE1 and SE10 are pretreated at very close severities but with different conditions. The mass loss of SE10 is slightly higher than for SE1 with the same moisture content before the pretreatment. It means that in spite of lower temperature the pretreatment duration influences the mass loss of the samples to a great extent. However the observed difference between these samples is not statistically significant.

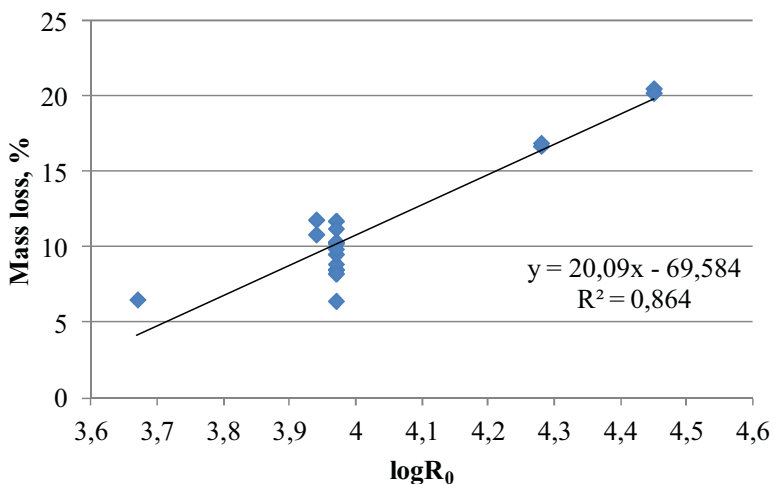


Fig. 1. Correlation between SEP severity and mass loss

The results of the used simple component fractionation vary depending on the severity of SE as seen from Fig. 2. Water soluble parts decrease significantly with the increasing severity. These parts include all residual hemicelluloses after the SE. The sum of water soluble parts and mass loss is equal to percentage values 29, 28, 22 respectively for SE1, SE2 and SE3. However the content of hemicelluloses in untreated grey alder wood is only 21 % (Zoldners et al. 2009). Our assumption is that the rest percentage may include extractives (5 %) and low molecular weight phenols and hydrolysed cellulose (Gravitis 1987).

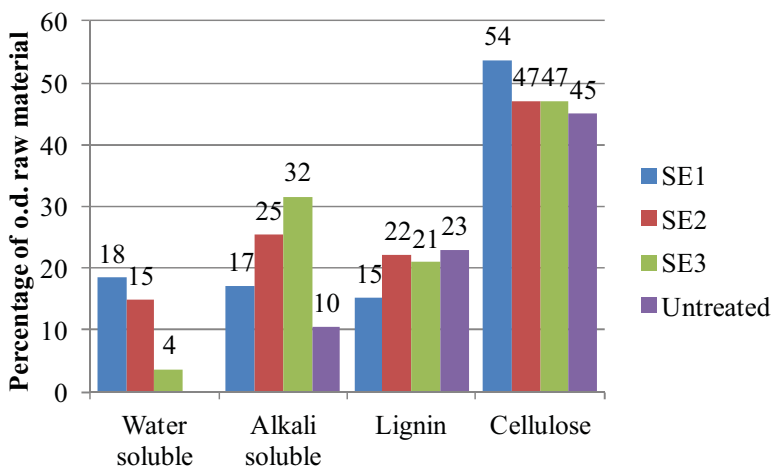
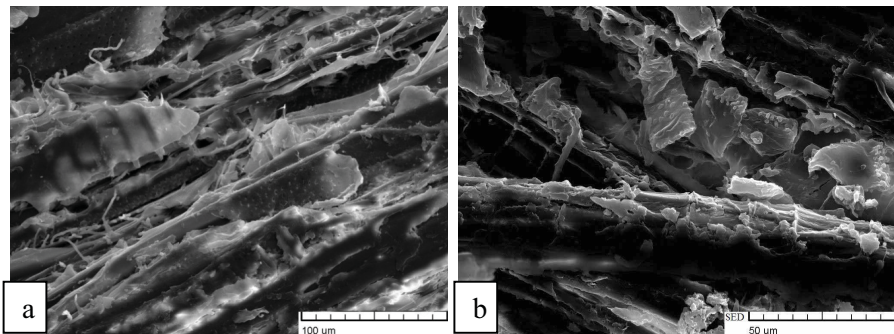


Fig. 2. Fractionation results of grey alder chips after SEP

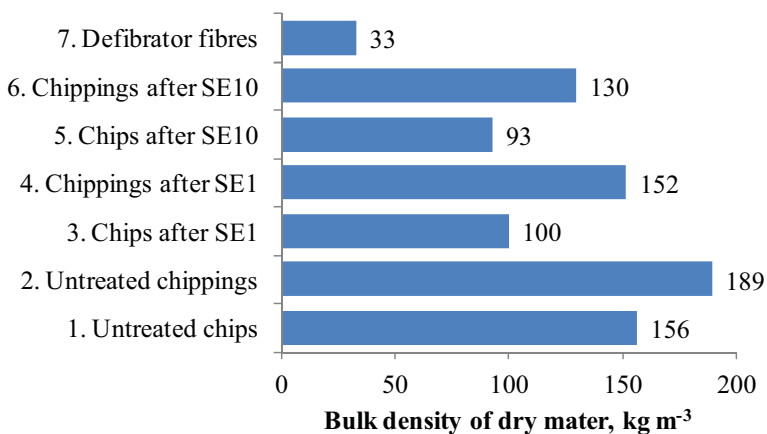
Alkali soluble parts increase with the increasing severity of SE and all the values are higher than of raw material (Fig. 2.). Extracted lignin from the alkali soluble parts contains less percentage values the rest parts staying in neutralisation solvent. The sample SE1 contains only 15 % of lignin while other samples contain similar amount to untreated chips. However sum of the contents of lignin and cellulose of all pretreated samples is very close to untreated sample. From this could be seen that lignin and cellulose are not degraded at used SE severities. Thus the used fractionation method is not effective only for lignin extraction from the SE1, however it effective for obtaining water soluble parts and the results agree with Heitz et al. investigations (1991).

Observations with SEM confirmed the chips microstructure changes after the SE. In Fig. 3. shown structure of untreated grey alder chip with its undamaged libriform and vessel elements (a); after the pretreatment the unit structure broken to fibre bundles and single fibres which are coated with caramel like lignin and residual hemicelluloses film (b). After the fractionation with water and alkali solution the fibre surface is smooth approving the mentioned substances on fibres before the fractionation. These observations agree with those found in the literature (Gravitis 1987; Shao et al. 2009).

The SE effect on the chips could be characterized also as changes of bulk density. The bulk density of the chips decreases after the SE1 at about 1.5 times but after the SE10 at about 1.7 times (Fig. 4.). In the case of the defined particles (chippings) the bulk density decreases only 1.2 times after the SE1 and about 1.5 times after the SE10. From this could be concluded that defibrillation level of the material during the SE depends on steam contact with the material duration more than on the temperature used. However, no one used the pretreatment conditions made the material bulk density similar to defibrator fibres obtained at MDF factory (Fig. 4.).



**Fig. 3.** SEM images of grey alder chips before (a) and after (b) SE



**Fig. 4.** Bulk density of grey alder particles before and after SE vs. defibrator fibre

## CONCLUSIONS

The most unstable constituents of grey alder wood degrade irreversibly during SE depending on the severity of the process and moisture content of raw material. The used fractionation of the pretreated biomass with room temperature water and following alkali solution results in about 100 % of lignin and cellulose extraction depending on the severity of SE. Observations with SEM confirm that during the SE the unit structure of the chips is broken to fibre bundles and single fibres coating its surfaces with caramel like lignin and residual hemicelluloses film. Defibrillation level of the material during the SE depends on steam contact duration with the material more than on the temperature.

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# BIO-BASED ADHESIVES AT LAMINATED VENEERS

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## ABSTRACT

Emissions from adhesives used for laminating wood-based composites has raised some environmental concerns, especially the use of formaldehyde in the formulation. The adhesives used need to form interface and interfacial bonds that are stable to different environmental climates. Tests have been carried out using lignosulfonates and tannin type compounds to determine if these can be used as adhesives. Result from this research show that it is possible but it needs further investigations.

Key words: biobased adhesive, lignin, lignosulfonates, tannin, laminated veneers

## INTRODUCTION

There are several methods of producing laminated wood. Navi and Sandberg (2012) have defined four product categories of laminated wood: lamination of veneers or boards for structural purposes, plane and cross-wise lamination of veneers i.e. plywood, manufacture of continuous laminated shapes, and forming and lamination of veneers simultaneously against a mould essentially for furniture and interior purposes. This paper is about shape stability of plane and cross-wise lamination of veneers glued with lignosulfonates or tannin.

Common adhesives for lamination of veneers are based on reactions of formaldehyde with phenol, resorcinol, urea, melamine or mixture thereof (Rowell, 2005). Urea formaldehyde (UF) resin; melamine urea formaldehyde (MUF) resin, or emulsion; polymer isocyanate (EPI) resin; polyurethane (PU) resin; and epoxy resin are examples of adhesives used for laminated bending of veneers.

Concerns around from bonded wood products have focused on formaldehyde but also other volatile compounds have been detected in different adhesive formulations. Heating increase the problem as it raises the vapour pressure of reactive chemicals. Isocyanates can react fast with compounds in human bodies. Both EPI and PU adhesives

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are containing isocyanates. (Rowell, 2005). Preparation and processing of epoxy also poses significant health risks in the form of e.g. allergies. There are also legal requirements which aim to depress levels of emissions especially free formaldehyde.

Environmental interest and rising cost of petroleum based adhesive i.e. synthetic adhesive has made bio-based adhesive of interest.

Most common bio-based adhesive are protein-based e.g. from “animal bones and hides, milk (casein), blood, fish skins, and soybeans”. Natural protein adhesives are not useful at high moisture levels. There are also bio-based adhesives possible to get from wood itself: tannin and lignin. Tannin are more reactive than phenol but more expensive. Limitations of tannin relative to synthetic adhesive are high viscosity, limited availability, and inconsistent source and therefore reactivity. Tannin has been used as adhesives in “composite (particleboard and medium density fibreboard) production, laminate and finger joint bonding”.

Lignin is available in large quantities at low cost. Lignin is a by-product of pulping processes for papermaking. Lignin is not as reactive as tannin with formaldehyde but can be modified. Lignosulfonates from sulfite pulping of wood have been found to be more useful for the production of reactive lignins (Rowell, 2005).

The aim of the work presented in this paper was to find out if it is possible to bond veneers together into a shape and moisture stable plan pressed laminate with lignin and/or tannin as adhesive.

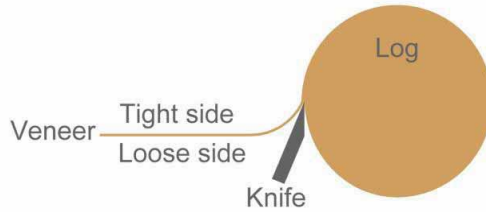
## MATERIAL AND METHOD

Three veneer with the middle veneer crosswise oriented have been glued into to a flat construction. Basic demand of the laminate was that it would be shape stable and useful in climates with high humidity without delamination in the bond line. Strength of the bond line has not been tested in this work.

In this work, beech (*Fagus silvatica* L.) peeled veneers with a size of 150x150x1.2 mm has been used. The veneers were conditioned at a relative humidity (RH) 20 % and temperature 20°C. The veneers thickness was made uniform by sanding before they were trimmed and conditioned. Used veneers have straight fibres.

Peeled veneer has one side that is commonly called the loose side and the opposite side is called the tight side. The loose side has fractures from the knife as it peels the veneer from the log, Figure 1. The most common method in laminated veneer products manufacturing is to have the orientation of the loose side inwards the glued assembly. Table 1 show the orientation of the three veneers as have been glued together.





**Figure 1.** Peeling of veneer.

**Table 1.** Orientation of the veneer, i.e. the direction of the loose side of veneer and fibre orientation in the plane where  $\perp$  means that the fibre orientation of the veneer is 90 degrees to the adjacent veneer (-).

Side of veneer (tight/loose) seen from above			Relative orientation between veneers in the plane of the assembly		
Top	Middle	Bottom	Top	Middle	Bottom
Tight	Tight	Loose	-	$\perp$	-

Lignosulfonates, tannin and mixture of lignosulfonates/tannin were used, Table 2. Adhesive spread of approximate 129 g/m<sup>2</sup> was used. The used lignosulfonates and tannin were commercial products. Lignosulfonates was from company Borregaard LignoTech and tannin was from company Kremer Pigmente.

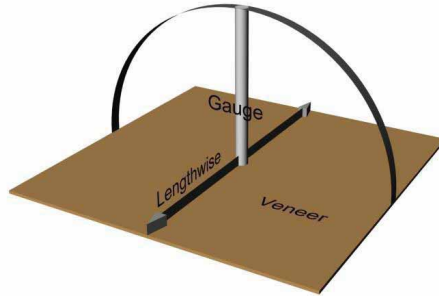
**Table 2.** Adhesives used in relation to the respective sample.

Sample	Adhesive
1	Lignosulfonates
2	Tannin
3	Lignosulfonates 75 %, Tannin 25 %
4	Lignosulfonates 75 %, Tannin 25 %

Pressing was made in a press with resistive heating of press plates. The temperature was 220 C, the contact pressure was 0.5 MPa and the pressing time was 20 minutes. The shape of the bow height and the condition of the bond line of the plan laminated was measured at different stage of climate cycling, Table 3. Figure 2 describe how the measuring of the bow height was determined.

**Table 3.** Climate cycling of the plan pressed samples.

Climate cycling	RH (%)	Temp (°C)	Time (days)
Phase 1, conditioning	20	20	19
Phase 2, conditioning	65	20	26
Phase 3, moistening	Above the dew point	30	34



**Figure 2.** The double arrow describes the lengthwise direction in the top veneer. The arc describes the positions on the veneer that measurement been based from. The cylinder describes the measuring of the bow height was made, in other words, in the centre of the veneer.

## RESULTS

All samples passed the first and the second phase of the conditioning without delamination in the bond line. At phase three in the climate cycling, there were partial delamination in the bond line at sample one, three and four. Measured bow height varied between samples, Table 4.

**Table 4.** Bow height (mm/150 mm) and controlled bonding line.

Sample	Time (days)		
	19	26	34
1	0	0	Partial delamination
2	0	0.5	0.5
3	0.4	0	Partial delamination
4	0.2	1	Partial delamination

## DISCUSSION

That it is possible to use lignosulfonates and tannin to bond veneer together is clear. If the high heat itself helps to activate the ingredients of the veneers is not clear but would be of interest to investigate. It also would be interesting to proceed to investigate the pressure parameters to find the brake lines of bonding. The climate cycling in phase 3 was supposed to be in a climate with relative humidity 90 % and temperature 20°C but the climate chamber did not work properly. In phase 3 just one sample of four passed regarding bond line. The other samples bond lines were partly delaminated.

## CONCLUSIONS

Research shows that it is possible to bond veneers together with biobased adhesive in form of lignosulfonates and tannin type compounds. The time and temperature for

optimum bonding has yet to be determined. Stability of the bond line to water and high humidity conditions also needs to be determined. Future research will concentrate in these areas.

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# THE PUZZLING TESTING OF THE DECAY RESISTANCE OF TIMBER

Venäläinen, M.<sup>1</sup>, Partanen, H. & Harju, A.<sup>2</sup>

## ABSTRACT

New recipes for timber preservatives as well as new timber modification methods are being invented in order to prolong the service life of wooden constructions in out-of-doors applications. There is a hurry to get the new products on the market but on the other hand, the function of the new preservative or modificate should be tested as reliably as possible before selling the end product to the consumers. The launching of a half-finished product in the market may even spoil the reputation of a promising innovation.

In this presentation we discuss the problems of getting balance between the testing time and costs, and the relevance of the results when normative durability tests EN252 (ground contact in field), EN113 (brown and white rot fungi *in vitro*) and ENV807 (soft rot in soil box) are used.

We also present preliminary data obtained by an innovative soil-contact testing box. This new-type soil box enables the measuring of strength loss that is taking place before the mass loss can be observed.

Key words: decay resistance of timber, decay testing

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# SELECTIVE SEED HARVEST FROM SEED ORCHARDS – A SHORT CUT TO SEEDLINGS WITH ABILITY TO PRODUCE DECAY RESISTANT SCOTS PINE HEARTWOOD

Harju, A.M.<sup>1</sup>, Venäläinen, M.<sup>2</sup>, Partanen, J.<sup>3</sup> & Kärkkäinen, K.<sup>4</sup>

## ABSTRACT

Heartwood is dead tissue rich of extractives. Those extractives, especially stilbenes pinosylvin and its' monomethylether, protect products made of heartwood against decay. Individual trees differ strongly from each other in the concentration of stilbenes in their heartwood, and thus also in the mass loss in decay tests. Great proportion of the measured variation in the concentration of stilbenes is inherited. Thus the progenies resemble their parents and siblings resemble each other. From the tree breeders point of view high heritability is useful. When desirable individuals are selected based on their measured values of stilbene concentration, high heritability determines that the individuals most probably have also desired genes in the selected trait. Breeding for chemical quality of Scots pine heartwood is very slow due to the late expression age of the trait, and because of the long rotation cycle. Thus we studied if the exploitation of the wide genetic variation in stilbene concentration could be speeded up by collecting seeds from those seed orchard clones that have been recognised to produce heartwood with high concentration of stilbenes. Material of this study consisted of seed orchard clones and their half-sib progenies growing in a field trial. This gave us an opportunity to compare the heartwood of the mothers to the heartwood of their children. In the presentation we discuss the results showing that selective seed harvest from good clones would be a possible way to hasten the production of seedlings having an inherited ability to produce durable heartwood when grown up.

Key words: heartwood timber, decay resistance, extractive concentration, *Pinus sylvestris*, seed orchard.

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# LIGNIN CONTENT ON HEMP SHIVES BY STEAM EXPLOSION CONDITIONS

Andzs, M.<sup>1</sup>, Veveris, A.<sup>2</sup> & Gravitis, J.<sup>3</sup>

## ABSTRACT

Hemp (*Cannabis sativa L.*), hemp shives are the short fibred inner woody core of the hemp plant. Hemp shives contains 65-80% of the stalk and are composed of libriform fibres who are high lignin content. Steam explosion (SE) auto-hydrolysis (also referred to as steam explosion pulping, flash auto-hydrolysis, or steam cracking) is a simple treatment of biomass (agricultural waste, logging residues, etc.) by saturated only steam without any additional reagents, usually at pressures up to 30 atmospheres. This is the only method how to produce lignin without any dash. Analogy if there is the most sever SE and more are destroyed hemp shives, the more lignin can extract. The aim is to definite the bulk volume, content of lignin in *Belobrzėsķy* hemp sort depending on the SE treatment factor, doing extraction with H<sub>2</sub>O and NaOH 0.4% and compare with out going content of hemp shives lignin.

Keywords: biomass, hemp shives, steam explosion, lignin, extraction.

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# VARIABILITY OF BIRCH BARK CALORIC CONTENT

Bosas, G.<sup>1</sup> & Petrauskas, E.<sup>2</sup>

## ABSTRACT

Renewable energy and the use of biomass in energy production promotes sustainable development and decreases the use of fossil fuels. Biomass, e.g. birch bark can be used in the production of heat and electricity, as well as being used as a biofuel component and novel product for the chemical industry. Efficient utilisation of biomass requires a high level of knowledge and the development of new processes to create a new way of thinking.

The aim of this research was to investigate relationships between bark thickness and along the stem bark caloric values of birch (*betula pendula*, *betula pubeskins*). This research was made on the bar of empirical data, collected in the front of central region of Lithuania. Data were taken from 10 trees. Totally were collected 133 sample discs, from bottom to top at measurement place: (0; 0,5 1; 1,3; 3; ...). Caloric values of samples were measured with ICA calorimeter c2000 and analysing with software statistica-8. Results from One-way analysis of variance, demonstrated that there is significant difference in bark caloric values taken in different place of stem. Bark caloric values with measurement, highest correlation with measure place on a stem ( $R = 0,485$ ) and bark thickness ( $R = -0,480$ ). Linear equations model were used compatibility approximate have relationships between bark caloric values and concerned factors. Mean of bark caloric value (dry matter) was measured 23,6 MJ/kg. Lowest caloric value were estimated of inner bark 20,11 MJ/kg, and highest-of outer bark 31,4 MJ/kg.

Key words: biomass, birch bark thickness, caloric value.

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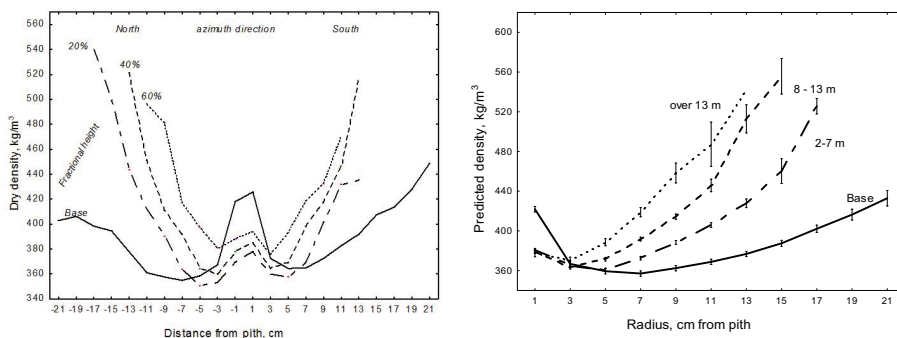
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# MODELLING A WOOD DENSITY PROFILE ALONG STEMS OF CULTIVATED NORWAY SPRUCE

Gjerdrum, P.<sup>1</sup> & Eikenes, B.<sup>2</sup>

## ABSTRACT

Density is one of the most basic quality traits, influential for wood processing and most applications. The aim of this paper has been to present a mathematical model for dry density variation along spruce stems. 85 mature spruce trees were sampled from afforestation plantations at the west coast region of Norway. 20 mm wide cubes of clear wood were extracted from the pith outwards in north and south directions at 10 equidistant heights along the trunk. Green and dry weight and volume were recorded. In this way, the 85 stems yielded 523 disks and a total of 6019 valid observation sets.



By examination of the observations (left graph), the following variable transformations were made:

$$H^* = \sqrt{H} \quad ; \quad R^* = \{Abs[R - (6.5 - 1.0 \cdot H^*)]\}^{1.45}$$

The resulting model for dry density (right graph) could then be estimated by general mixed model GLM, setting tree number as random factor:

$$DryDens = 352.6 + 1.48 \cdot R^* + 1.60 \cdot H^* + 1.01 \cdot R^* \cdot H^*$$

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Overall  $R^2 = 0.64$ , of which between stem variation  $s = 35 \text{ kg/m}^3$  accounts for two thirds. The model standard error is  $32 \text{ kg/m}^3$ ; however, the model fails to identify the most extreme density values. The most important results are the quantitative, mathematical form of the model, and the identification of a general density minimum at a certain distance from the pith.

Key words: Dry density, density equation model, radial density variation

# EVALUATING THE DIMENSIONAL STABILITY AND DEFORMATION OF PLYWOOD PANELS

Paajanen, O.<sup>1</sup>, Mäki, V-M. & Kairi, M.

## ABSTRACT

Plywood is a panel product that is manufactured by gluing thin sheets of wood, called veneers, together under heat and pressure. The final product is used in variety of applications, often in construction. Plywood is a comparatively high value product, especially when it is used in demanding applications where the quality of the product is very important. Plywood has good strength properties relative to its weight. However, the deformation, e.g. the warping of plywood is an issue. Warping and other dimensional stability problems are very typical in thinner plywood panels, especially when they are subjected to variable humidity conditions. The stability of plywood is very important criterion for the use of plywood in some special applications. There are some standards that are used to determine these properties in panel products, e.g. EN 13647 (Wood and parquet flooring and wood panelling and cladding. Determination of geometrical characteristics) and EN 910 (Wood and parquet flooring and wood panelling and cladding. Determination of dimensional stability). The dimensional stability tests are based on humidity cycle testing. This poster discusses the standards, measurement systems and also the problems associated with the dimensional stability and warping of plywood. Some measurement data and figures which describe the problem will also be included.

Key words: plywood, wood based panels, deformation of plywood, dimensional stability

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# PLYWOOD MANUFACTURE WITHOUT ADHESIVES

Ruponen, J.<sup>1</sup>, Rautkari, L.<sup>2</sup> & Hughes, M.<sup>3</sup>

## ABSTRACT

Plywood is commonly produced from rotary-cut veneers that are cross-laminated after adhesive spreading, then cold-pressed and finally hot-pressed. However, there is a less known way to manufacture plywood without any external adhesives where phenomenon called auto-adhesion is applied. The method is known also as self-bonding or binderless bonding of wood. Auto-adhesion requires higher temperature, pressure and moisture content of the veneers compared to ordinary plywood production. The phenomenon itself is already known and successfully applied since 1930s at hardboard production, yet with a significantly smaller fraction size. During last decade, the topic was studied in Sweden and Japan with several wood species. This method enables plywood manufacture without external adhesives and the process could be fully free from fossil resources. At Aalto University the self-bonding of wood is being studied since 2010 and both Norway spruce (*Picea abies*, L.) and birch (*Betula pendula*, L.) have resulted successful adhesion. According to these studies as well as the previous studies completed in Sweden and Japan the self-bonded plywood suffers from delamination when exposed to moist conditions. This problem occurs also in the bond line achieved through linear friction welding which is another bond type representing auto-adhesion. According to the studies completed at Aalto University a certain kind of thermal treatment enhances greatly the bond stability in moist conditions. Most probably this enhancement relates to lessened equilibrium moisture content and increased hydrophobicity as well as improved dimensional stability caused by lessened internal stresses. However, the process is not fully controlled nor understood and it needs improvements in controlling the internal vapour pressure within the lay-up to hinder blow ups as well as optimise the process pressure and temperature as well as the initial moisture content of the veneers.

Key words: auto-adhesion, binderless bonding, veneer, plywood, self-bonding.

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# CHARACTERISTICS OF THE HEMP FIBER MODIFIED PLYWOOD

Upitis, G.<sup>1</sup> & Dolacis, J.<sup>2</sup>

## ABSTRACT

With the growth of the environmental pollution and the usage volumes of traditional natural resources, more and more attention is paid to the use of environmentally friendly materials. Scientists of the world are working on acquiring new materials and improving the characteristics of the existent ones. New material fusions have been created and as a result composite materials with better or even completely different characteristics have been developed. During the production of the new generation source materials and equipment, a lot of attention is paid to the environmental impact of the process and to the recycling of the products. More than 100 years ago the invention of the plywood peeling and later also carving hardware revolutionized the woodworking sector. It opened the way to the production of a new material – plywood. With the ever growing usage of plywood in the national economy, the necessity arises to increase its physically mechanical characteristics and reduce production costs. Plywood with different coatings that change the mechanical characteristics of its surface has already been developed and introduced into production. One of the ways for development is to create composite materials – supplement plywood with hemp fibre fabric.

The paper reflects the results of a research on a five-layer birch plywood composite material with hemp fabric reinforcement. There is a research on the optimum proportions of the glues, gluing parameters and their influence on the final product. Tests of the physically mechanical characteristics of the material patterns have been carried out and the acquired results have been processed and compared with the existent standards. The average strength index of the material has increased by 13.2%, and the guaranteed index has increased by 42.5% compared to the control patterns.

A new use for the hemp fibre has been found. As a result a new, environmentally friendly material with high physically mechanical characteristics has been developed, which and can be used to produce plywood and bent glued wood constructions.

Keywords: plywood, hemp, plywood composite material.

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# HOW TO EXPLORE THE USER EXPERIENCE OF WOOD IN INTERIORS

Vahtikari, K.<sup>1</sup>

## ABSTRACT

Department of Forest Products Technology at Aalto University, Finland, is interested in to explore the user experiences of wood in interiors and the importance of material choices regarding the perception of thermal comfort. There are three test houses in the university campus that can be used for user experience tests. The air flow, relative indoor humidity and indoor temperature can be monitored and controlled. The houses are equal in size, but differ in interior materials. Interior walls and ceiling are made of gypsum board, plywood or solid wood panels. The floor in all three houses is made of solid wood.

This poster describes different test set-ups for exploring user experience of wood in interiors.

Key words: user experience, wood interiors, thermal comfort.

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# ADHESION AND WETTING PROPERTIES OF CHEMICALLY AND THERMALLY MODIFIED WOOD

Vitosytė, J.<sup>1</sup>, Grigaliauskienė, I.<sup>2</sup> & Ukvalbergienė, K.<sup>3</sup>

## ABSTRACT

Wood is not very durable in long terms when is exploited outdoors. Its exposure to moisture and UV radiation leads to changes in measurements, shape and colour as well as to the deterioration of adhesive properties, photodegradation or the development of cracks. Moreover, it is gradually destroyed by nematodes, fungi and bacteria. In order to enhance the durability of wood, it undergoes various modifications (chemical, thermal) or is covered with different polymeric materials. Due to the low compatibility of wood surface and polymer, extremely high hygroscopicity, porosity, anisotropy and surface properties of wood, when exploiting outdoors, adhesion between wood substrate and polymer rapidly and intensively decreases and wood finishing material ceases to function as protective element. When modified thermally or chemically, wood chemical structure changes and wood becomes more hydrophobic, therefore poorly absorbs water. This advantage becomes limitation when modified wood is glued or finished with synthetic polymers: surface wetting, coating spreading on the wood substrate and bonding between wood surface and polymer are poor. Consequently, during the exploitation time, wood coating flakes off. Still it is a big problem in the market although the demand for modified wood is high and will only increase in the future. It is not yet fully understood, how changes the adhesion mechanism when modifying wood. Also it is important to understand the wetting processes. Therefore in this research the wetting and adhesion properties of thermally and chemically modified wood finished with water-based acrylic coatings were studied. For this research defectless oak wood samples with dimensions of 50 x 50 x 15 mm were used. All samples were distributed into 4 groups – heat treated (160°C and 220°C), ammonia modified and unmodified as a reference. Wood surface roughness, pH, wetting angle using sessile drop method and adhesion strength (according standard EN ISO 4624:2000) were evaluated.

Keywords: heat treatment, adhesion, chemical treatment, contact angle

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# RESEARCH OF SOME MECHANICAL PROPERTIES OF BIRCH PLYWOOD I-BEAMS

Lipinskis, I.<sup>1</sup>, Spulle, U.<sup>1</sup> & Tuherm H.<sup>1</sup>

## ABSTRACT

Plywood is one of the well known wood panel materials their panel sizes are standardized. After cutting plywood according to the required dimensions of the client needs production residues extent that further use is limited and reason is dimensions. In case to find application for these production residues, could significantly increase the use of plywood materials and it could be the step towards to non-waste production of plywood. Research of plywood physical and mechanical properties was done before and the resulting technology production residues specification was chosen for I-beam construction and research was carried out.

Research shows that plywood I-beam ultimate bending moment and stiffness values are higher than other manufacturers offered beams this parameter values used in the manufacture LVL (Laminated Veneer Lumber) and wood materials. Research shows that in several cases moment of resistance and stiffness in bending of birch plywood I-beam are higher than other producer's characteristics. Also worth noting that in all cases tested beam width, depth and cross-sectional area is slightly smaller than the beam of other producer's sizes. Characteristic for the tested beams is considerably below the other, is the mass.

Key words: birch plywood, I-beam, strength, modulus of elasticity, stiffness.

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# PREDICTED GAIN IN WOOD HARDNESS OF LITHUANIAN POPULATIONS FROM SCOTS PINE, SILVER BIRCH AND BLACK ALDER PROGENY TESTS

Baliuckas, V.<sup>1</sup>

## ABSTRACT

According to National Forest Inventory data forest land area has increased by 1.9% (125.000 ha) in Lithuania. Softwood deciduous forest land increased most, especially areas of birch species, followed by aspen and black alder. Scots pine stands occupy 35.3%, birch – 22.2%, black alder – 6.8% (statistics year 2011).

It is pointed out in the National Forestry Sector Development Programme that up to 70 percent of all forest reproductive material has to be raised using seeds from second generation or higher-level seed orchards. Intense use of established field test trials of forest tree species for breeding purposes becomes very important in this context.

Scots pine, silver birch and black alder half-sib families of plus trees were tested for wood hardness in the series field trials at ages 30, 13 and 13, accordingly. The trials consist of 7 populations and 140 families of Scots pine, 24 populations and 100 families of silver birch, 17 populations and 85 families of black alder. Wood hardness was tested by using Pilodyn 6J. Mean value of Pilodyn pin penetration for pine families was 19.3 mm, for silver birch – 23.4, for black alder – 22.0. The average difference between family mean estimates for pine was 4.5 mm, for silver birch – 4.0, for black alder – 4.2.

The same complex selection index was used for each species, including wood hardness, height, diameter, stem straightness, branch quality and survival estimates. Height was given an economic weight of 1.5 and wood hardness – 1.3. Such index and selection of the best 15 families and 30 trees in selected families for the higher-level seed orchards are typically used in practical breeding work in Lithuania. Wood hardness and tree height traits were the most heritable. Individual heritability for Scots pine wood hardness was 0.30 and family heritability was 0.65. For silver birch similar estimates were 0.61 and 0.75, for black alder – 0.78 and 0.86. The average genetic gain from all provenance regions in wood hardness (when selecting 15 best families) was

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estimated for Scots pine 0.5%, silver birch – 3.1%, black alder – 4.8%. The same estimates of genetic gain when selecting 30 best trees in selected families were: 1.6%, 5.7% and 7.4%. If only height and wood hardness were used in calculation of breeding values, then genetic gain for the last trait would be 1.5-2.0 times higher.

Gene resources conservation and forest tree breeding is joint in Lithuania. Study results indicate that it is economically adjusted to start diversified breeding in some widespread conifers and broadleaves.

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