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Proceedings of the 7th meeting of the NORDIC-BALTIC NETWORK IN WOOD MATERIAL SCIENCE & **ENGINEERING (WSE)** 

October 27-28, 2011, Oslo, Norway

Edited by Erik Larnøy and Gry Alfredsen



NORWEGIAN FOREST AND LANDSCAPE INSTITUTE



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Cover Photo: Spiral pattern of reaction wood in spruce. Photo: Andreas Treu, Skog og landskap Norsk institutt for skog og landskap, Pb. 115, NO-1431 Ås

# PREFACE

The Nordic-Baltic Network in Wood Material Science and Engineering (WSE) was established in 2004 by the Nordic Forest Research Cooperation Committee (SNS). The field of the network, wood science and engineering, covers wood-water relations, wood durability, wood modification, wood mechanics, wood composites, engineered wood products, eco-efficient wood based products, wood engineering, wooden buildings, and use of machines and constructions.

The first six 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)

The network is now an established platform for the exchange of knowledge in the fields of wood science and engineering in the Nordic - Baltic region. It is an arena that makes it possible for experienced researchers and students to meet and coordinate research, and stay up to date on the latest research.

During the past six years the network has constantly been growing, resulting in attracting 80 researchers from ten countries in 2011. The present proceedings contains 39 papers and 10 posters.

The section of Wood Technology at the Norwegian Forest and Landscape Institute has the honour to host the meeting in 2011. 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  $300 \in$  is this year presented by our sponsor JOTUN

Ås, October 2011

Erik Larnøy



# SUMMARY

This report presents presentations and summaries of posters presented during the conference "7th meeting of the Nordic-Baltic Network in Wood Materials & Engineering". The conference was held Oct. 27 to 28, 2011 in Oslo, and gathered around 70 participants from 9 countries. Norwegian Forest and Landscape institute hosted the event.

# SAMMENDRAG

I denne rapporten presenteres foredrag og sammendrag til plakater som ble presentert under konferansen "*7th meeting of the Nordic-Baltic Network in Wood Material & Engineering*". Konferansen ble arrangert 27-28 oktober 2011 i Oslo, og samlet ca. 70 deltakere fra til sammen 9 land. Norsk institutt for skog og landskap var vertskap for arrangementet.

Key Words: wood composites; eco-efficient wood based products

Nøkkelord: tre og fuktighet, treholdbarhet, tremodifisering; tremekaniske egenskaper, trekompositter, miljøeffektive trebaserte produkter

wood-water relations; wood durability; wood modification; wood mechanics;

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# NATURAL DURABILITY OF WOOD IN NORWAY – RESULTS AFTER EIGHT YEARS ABOVE GROUND EXPOSURE

Evans, F.<sup>1</sup>, Alfredsen, G.<sup>2</sup> & Flæte, P.O.<sup>1</sup>

#### ABSTRACT

Some of the most common Norwegian wood species were tested in a Double layer test in South East Norway. After eight years of exposure the highest decay rating ( $\geq$ 3) was found in Scots pine sapwood, Norway spruce, alder, birch and aspen. Two wood types had decay rate  $\leq$ 1: Scots pine heartwood and cedar. Wood moisture was logged and compared with precipitation during a two month period the second year of exposure. Scots pine sapwood had higher wood moisture content than Norway spruce, and a good correlation was found between precipitation and wood moisture content. When comparing similar materials exposed at three different geographical locations in Southern Norway, the samples exposed in Bergen had higher decay rating than samples exposed at Ås and Oslo.

Key words: Norway, natural durability, geographical variation, above ground testing

#### INTRODUCTION

In Norway exterior wood structures have traditionally nearly exclusively been made of treated or untreated Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*). In recent years there has been a tendency that other tree species, like various domestic hardwoods and imported species have been used in exterior above ground applications - use class 3 (EN 335-1, 2006), often without any surface treatment. One of the principal reasons for the increased interest in using "new" species is that the different visual appearances of different species of wood offer a broader range of aesthetical elements in architecture, and thereby enable customised solutions in house building. Another aspect is the increased focus on utilising natural durability as an alternative to traditional wood preservation.

Natural durability of wood is determined by the European standard EN 252 (1989) for specimens in ground contact and EN 113 (1996) for basidiomycetes in the laboratory, but no standard test are included for above ground conditions. For above ground conditions, the European technical standard CEN/TS 12037 (2003) and EN 330 (1993)

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are used to determine the durability of treated wood. In addition, a range of nonstandard tests are used, among them are the Double layer test (Rapp and Augusta 2004). An overview of testing and evaluation of natural durability of wood in above ground conditions in Europe is published by Råberg et al. (2005). Among their conclusions are that the most important factors for fungal establishment on the surface and within wood are the moisture content, the surrounding temperature, and the relative humidity.

The aim of this paper was to evaluate the natural durability of Norwegian wood species in above ground exposure.

#### MATERIAL AND METHODS

#### Wood material

The wood species used in this project are the most common Norwegian wood species able to give sufficient dimensions for sawn wood. In addition two imported reference species were included: Siberian larch and Spanish cedar. All wood materials are listed in Table 1, 20 test specimens were used for each wood species.

Abbreviation	Latin name	Common name	
	Hardwoods		
Alder	Alnus glutinosa/Alnus incana	Alder/ Grey alder	
Birch	Betula pendula/Betula pubescens	Silver birch/Downy birch	
Aspen	Populus tremula	Aspen	
Oak	Quercus petraea/Quercus robur	Sessile oak/Pedunculate oak	
	Softwoods		
Spruce	Picea abies	Norway spruce	
Pine s	Scots pine sapwood		
Pine h	Pinus sylvestris	Scots pine heartwood	
Pine h-n	Pinus sylvestris	Scots pine heartwood (narrow annual year rings)	
Sitka	Picea sitchensis	Sitka spruce	
	Imported species		
Cedar	Cedrela spp.	Spanish cedar	
Larch	Larix sibirica	Siberian larch	

**Table 1.** Wood species used in the project: abbreviation, Latin name and common name.

#### **Test methods**

The Double layer test (Rapp and Augusta 2004) was used for above ground exposure. The samples rested on inert aluminium frames. The test site was the roof of the Norwegian Institute of Wood Technology's 8 floor building at Blindern, Oslo. The test started in 2002 and has been evaluated annually (with the exception of 2006). For the comparison between test sites, evaluation data after six years were compared with six year double layer data from field tests established in Ås and Bergen in 2004 (Flæte et al. 2008, 2011). The samples in Ås and Bergen are from the same batch of materials. Decay was evaluated according to the rating system of EN 252 (0 = no decay, 4 = failure). Wood moisture content was logged in a two month period the second year of exposure in three Scots pine sapwood and three Norway spruce specimens.

#### **RESULTS AND DISCUSSION**

In Fi.1 mean decay ratings after eight years is presented. Within this period none of the wood species reached failure (rating 4) for all test specimens. A mean decay rate of  $\geq 3$ 

after eight years was found in Scots pine sapwood, Norway spruce, alder, birch and aspen. Two wood materials had  $\leq 1$  decay rate: Scots pine heartwood and cedar. The remaining species/wood types had a rating between 1 and 2.



**Fig. 1.** Mean decay ratings (0 = no decay, 4 = failure) after eight years of exposure above ground (Double layer test) at Blindern, Oslo. Variation is given as standard deviation.



**Fig. 2.** Wood moisture measurements in (a) Norway spruce and (b) Scots pine specimens (yellow, pink and blue) comapred with precipitation (light blue) measurements.

In Fig. 2 wood moisture content and precipitation in a two month period the second year of exposure is presented. The wood moisture content correlates well with the precipitation, this includes a slight time lag. Norway spruce showed much lower wood moisture content then Scots pine sapwood.

In Fig. 3 mean decay rate (rating/year) from all eight years is presented. During the exposure period there have been no huge shifts in durability ranking between species. Scots pine sapwood has throughout the test period the highest decay. No significant differences was found between the two types of Scots pine heartwood. One should be careful to interpret too much from ratings below 2. Field evaluations are rather subjective and climate and moisture conditions might influence the evaluation results, one obvious example is the birch results after six years. In this study we have done independent evaluation, not looking at previous year's results during evaluation.

In Fig. 4 the decay rating after six years in Oslo is compared with similar materials exposed at Ås and Bergen. Oslo and Ås are located in the same region in Eastern Norway with similar climatic conditions, while Bergen is located in the humid coastal region in Western Norway. Generally, Bergen has the highest decay rating. Between Ås and Oslo there is no obvious trend, it varies between species. Norway spruce had surprisingly similar rating between the test sites. It has to be noted that the test sites in Bergen and Ås is exposed above soil, while the test setup in Oslo is located on a roof. This might cause some of the variation between Oslo and Ås.

Still knowledge is lacking, both in Norway and in general, about species diversity, colonisation and succession patterns in different wooden materials used outdoors. Identifying fungi with traditional methods, like agar plate isolation, is time consuming and not very accurate. Molecular methods, e.g. PCR and sequencing, is an objective approach for species identification, and it does not require mycological skills (Råberg et al. 2005).



Fig. 3. Mean decay rate (rating/year) for (a) softwoods and (b) hardwoods.



**Fig. 4**. Mean decay rating after six years of exposure in Double layer test showing geographical variation between three different localities in Norway: Bergen, Oslo and Ås. Softwoods are illustrated in (a), hardwoods in (b). Variation is given as standard deviation.

To improve our knowledge about wood protection and to utilize wooden materials in an optimal way we need to improve our utilization and evaluation of field trials. Logging temperature and moisture will provide important information about how climate affects the service life of outdoor wooden constructions. Evaluation should also be taken one step further, not only using the traditional decay rating system. Råberg et al. (2005) concluded that strength tests are the most sensitive for decay detection, but neither strength tests nor identification of fungi responsible for the decay are included in the standards of above ground durability in field tests.

b

#### CONCLUSIONS

- After eight years of exposure Scots pine sapwood had the highest decay rating, Scots pine heartwood and cedar the lowest decay rating.
- Wood moisture content was higher during the measuring period in Scots pine sapwood than in Norway spruce.
- When comparing similar materials exposed at three different locations in Southern Norway, the samples exposed in Bergen had the highest decay ratings.

#### ACKNOWLEDGMENT

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# FUNCTIONAL GENOMICS OF WOOD DEGRADATION – A PROJECT SUMMARY

### Alfredsen, G.<sup>1</sup>

#### ABSTRACT

Modified wood can provide protection against a range of wood deteriorating organisms. Several hypotheses have been put forward for the mode of action against wood decaying fungi, including inhibition of action of specific enzymes, but they still need further testing. This paper summarizes results from a project focusing on molecular studies of fungal colonization in modified wood. The focus has been on furfurylated wood, but also thermally modified and acetylated wood has been studied.

Among the main finding was that wood modifications have an effect on the exploitation face of both brown and white rot colonization, but not on the exploration face. As already reported in a range of papers wood modification effects the wood moisture content, and this was confirmed within this project. New information was gathered about the effect on gene expression. Even before any mass loss was detected, differences in gene expression were measured. Within an eight week period, genes related to oxidative metabolic activity of P. placenta generally was higher in furfurylated wood compared to untreated Scots pine. Carbohydrate metabolism related expression varied. A similar comparison was done, but with longer incubation time and also including thermal modification and acetylation. In the beginning of the incubation of all treated wood samples, the genes coding for oxidative metabolic activity had higher expression levels than the untreated control. In the end of the incubation most of these genes were less expressed than in the untreated control. The genes used for carbohydrate metabolism and the alcohol oxidase showed a significant decrease after 14 weeks of incubation. At the same time an increase in gene expression of an enzyme putative involved in lignin decomposition was detected. It was also shown that the use of molecular methods in field trial evaluation can contribute with important additional information to the standard evaluation methods.

Key words: fungal colonization, gene expression, mode of action, quantitative real time PCR, wood modification,

#### INTRODUCTION

Environmentally more benign methods are warranted for wood protection. A range of studies the last decade have shown that modified wood can provide protection against a range of wood deteriorating organisms, including decay fungi. Wood modification involves the action of a chemical, biological or physical agent upon the material,

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resulting in a desired property enhancement during the service life of the modified wood (Hill 2006). An understanding of the mechanisms utilized by decay fungi when exposed to modified wood is important for further optimisation of new modified wood products. The mode of action of modified wood systems can be explained by three hypotheses put forward by Hill (2006): (1) The first scenario is the inhibition of action of specific fungal enzymes. The hydroxyl groups in the cell wall and/or in the lumen are substituted with other groups, causing the enzymes to no longer recognize the substrate. (2) Secondly, the equilibrium moisture content is lowered in modified wood, and therefore it is harder for fungi to access the moisture required for decay. (3) Thirdly, modification could cause blocking of cell wall micropores, and this fact lowers the substrate accessibility for decay fungi.

Application of the new molecular procedures to questions concerning the decay of wood and biocide breakdown by the wood decay fungi and associated microbial communities lags behind many fields of biology (Diehl et al. 2008). The use and new possibilities of molecular tools within the field of wood protection has been summarized by several authors (e.g. Diehl et al. 2008, Gelhaye and Morel 2009). To improve laboratory tests, biomarkers of wood degradation need to be developed taking into account the complexity of the wood composition and of the degradation mechanisms (Gelhaye and Morel 2009). Another challenge is that the exact mechanism of brown rot decay still is hypothetical and controversial (Kang et al. 2009a). The molecular tools developed within microbiology allow us to study gene expression, protein presence and enzyme activity. Few studies have been published so far, but one example is Kang et al. (2009b). They studied gene expression of selected decay enzymes produced during biodeterioration of three wood types. Among the findings was that it appears that ACQtreated wood do not repress the production of the decay enzymes by the fungus but does inhibit the effectiveness/access of these enzymes on the modified substrate. Results from the study indicate that different resistant woods have different effects on the microbial communities and its enzymatic activities during decay.

It is worth to keep in mind that no single research technique can answer all questions about the decay of wood, we need to gather small pieces of the puzzle using different approaches (Diehl et al. 2008).

The aim of this paper is to summarize the results related to wood protection from the project 'Functional genomics of wood degradation: strategies used by decay fungi against wood protection systems and natural host defence compounds'.

#### SUMMARY OF RESULTS

#### Comparing DNA content in modified wood

In Pilgård et al. (2010) quantitative real-time polymerase chain reaction (qPCR) was used to profile the DNA amounts of *T. versicolor* (L.) Lloyd (strain CTB 863A) during colonization of treated *Pinus sylvestris* (L.) sapwood. The wood modifications used were acetylation, furfurylation, and thermal modification, samples were harvested after 2, 4, 6 and 8 weeks. The traditional wood preservatives Cu-HDO and CCA were used as references. The maximum levels of fungal DNA in control specimens occurred after 8 weeks. For all wood treatments, the maximum fungal DNA level was recorded after an incubation period of 2 weeks, followed by a decline until the end of the trial at 8 weeks.

The observed decline in fungal DNA amount after 2 weeks of incubation probably reflected the inability of the mycelia to establish a wood exploitation phase on the treated wood. Fungal colonies have been found to exhibit autolysis of older parts of the mycelia, particularly when growing on nutrient-poor media (Olsson 2001). A longer incubation time was suggested for new studies to be able to learn more about colonization in modified wood. Thermally modified wood had the highest and furfurylated wood the lowest levels of total T. versicolor DNA throughout the test period. This trend was, however, not significantly proved. The possible lower amount of fungal DNA in furfurylated wood after 8 weeks might be owing to polymerization of the furfuryl alcohol in wood and this led to a physical blocking within the wood cell wall. Venås (2008) hypothesized that the reduced accessibility of carbohydrates in furfurylated wood is most probably owing to cell wall bulking. The observed low colonization level in furfurylated wood might lead to the conclusion that T. versicolor is not able to utilize the furfuryl alcohol polymer as an alternative carbon source. The most probable explanation of the levels of fungal DNA in thermally modified wood is the heat inflicted destructions in the wood cell walls. The heat also degrades hemicelluloses to a greater extent than other macromolecular components (Shafizadeh and Chin 1977), resulting in easier access to lignin for the white-rot fungus T. versicolor. Acetylation is a modification of the OH groups in the cell wall without polymerization and the treatment does not damage the cell wall. Acetylation falls between the destructive thermal modification and the blocking furfurylation concerning the severity of various wood modifications. Consistently, the colonization level of T. versicolor in acetylated wood was more pronounced than that observed in furfurylated wood. Other factors that might contribute to differences between the different modifications include pH and moisture content, but also possible differences in virulence in the different Petri dishes in the test. For the preservative-treated woods, Cu-HDO showed the lowest level of fungal DNA throughout the experiment, indicating that exploratory hyphal growth is limited owing to the phytotoxicity of the treatment. The other treatments did not inhibit the exploratory hyphal growth phase.

In Schmöllerl et al. (2011) data from mass loss, qPCR and qRT-PCR were used for profiling growth dynamics and gene expression of Postia placenta (Fr.) M.J. Larsen & Lombard (strain FPRL 280) in different wood substrates through different stages of decay. P. sylvestris sapwood was used for the following treatments and modifications: CCA, furfurylation, thermal modification and acetylation. The paper presents results from different time intervals, 2, 14 and 26 weeks. As already reported in a range of papers wood modification effect the wood moisture content, and this was confirmed within this study. The highest mass loss and the highest fungal DNA content were found in the control samples while acetylated wood had the lowest mass loss and fungal DNA content. The data from all treatments reflected a close relation of mass loss and fungal DNA content. This confirms earlier finding, e.g. Eikenes et al. (2005). Except for the CCA treated wood, the DNA content decreased after 14 weeks of incubation, this emphasizes the hypothesis of autolysis and/or reallocation within the hyphae of the fungus (Olsson 2001, Pilgård et al. 2010) after incubation on a nutrient poor substrate. In the CCA treated reference, the growth of the fungus seemed to start after a lag phase. This could be a consequence of buildup of tolerance to the preservative, but this hypothesis has to be proven by longer incubation. Anyway, it is no surprise that the maximal DNA content in the treated samples is lower than in the untreated control samples, confirming a protective function of all investigated wood treatments. In acetylated wood the DNA content decreased already after 2 weeks of incubation,

indicating a low availability of nutrients in the wood for *P. placenta*. This is consistent with the findings in Pilgård et al. (2010) using *T. versicolor* and 8 weeks of incubation.

#### Gene expression in modified wood

In Alfredsen and Fossdal (2010) gene expression of the brown rot fungus *P. placenta* was monitored after 2, 4 and 8 weeks of colonization in furfurylated *P. sylvestris* sapwood and in untreated control samples. The main finding was that genes related to oxidative metabolic activity generally was higher in furfurylated wood compared to untreated Scots pine. Carbohydrate metabolism related expression varied. For one endo-glucanase and two  $\beta$ -glucosidases the expression was lower in furfurylated wood compared to untreated control, while for one glucoamylase and one glucan 1,3b glucosidase the expression was higher in furfurylated wood. The four cytochrome P450 tested, involved in breakdown of toxic compounds, gave inconsistent results between furfurylated and untreated control samples. Phenylalanine ammonia lyase and cytosolic oxaloacetase gave higher expression in control than in furfurylated samples.

Generally, the increase in gene expression of all investigated P. placenta genes in Schmöllerl et al. (2011) was highest in CCA treated wood, and this suggest that the fungus is transcriptionally active despite not actively growing during the first 2 to 14 weeks on CCA. In the beginning of the incubation period, the genes coding for oxidative metabolic activity (Lac1 and AlOx) in treated wood samples had higher or similar expression rates compared to untreated control. The need for oxidative enzymes for the degradation of wood modifications and a stress response are possible interpretations for higher gene expression which were also described by Alfredsen and Fossdal (2010). In the end of the incubation, most of these genes tended to be less expressed in modified wood than in the non-treated control. The need for oxidative enzymes for the degradation of wood modifications and a stress response are possible interpretations for higher gene expression which were also described by Alfredsen and Fossdal (2010). In this investigation, the analysed time period of decay was longer, showing a clear decrease in gene expression of alcohol oxidase (AlOx) in modified wood after 26 weeks of incubation. According to the stress response interpretation the following reduction of gene expression could be explained by an adaption of P. placenta to the modification. If the genes were used for the neutralization of modifications, the later decrease of the gene expression could account for an effective reduction of inhibitory wood modifications, which results in a better access to sugar containing nutrients. The genes used for carbohydrate metabolism (EGlu3, Gamy) and the alcohol oxidase (AlOx) showed a decrease after 14 weeks of incubation for the different wood modifications. At the same time an increase in gene expression of a putative lignin degrading enzyme (MPOX) was detected. The combination of these two effects could be interpreted as a shift towards another metabolic pathway or reflect stress associated with fungal cell death and failed colonization attempt on treated samples.

#### Basisiomycete colonization in field stakes using qPCR.

The aim of Pilgård et al. (2011) was to evaluate (qPCR) as a tool for investigating details of the colonization pattern of basidiomycete decay fungi in wood samples after 6 years of soil exposure. Samples of *P. sylvestris* (heartwood without treatment), furfurylated *P. sylvestris* sapwood and Cu-HDO treated *P. sylvestris* sapwood was used.

The qPCR method based on basidiomycete DNA content in the wood had the highest sensitivity, while the ergosterol assay was more sensitive than the chitin assay. Visual rating was compared with laboratory analyses and was found to be correlating well with qPCR. This study demonstrates that qPCR in combination with microscopy provides relevant data about basidiomycete colonization in wooden field test materials.

#### CONCLUSION

- The maximum *T. versicolor* DNA level was recorded after 2 weeks, followed by a decline until the end at week 8 when comparing furfurylated, thermally modified and acetylated wood with control and two wood preservatives. Control samples had a gradual increase throughout the test period. One interpretation is that the fungus is able to colonize, but not utilize the modified within this timeframe.
- *P. placenta* was able to start causing mass loss in thermally modified and furfurylated wood after 14 weeks.
- Within an eight week period, genes related to oxidative metabolic activity in *P. placenta* generally was higher in furfurylated wood compared to untreated Scots pine sapwood. Carbohydrate metabolism related expression varied.
- Generally, expression of the investigated *P. placenta* genes we highest in CCA treated wood. In the beginning of the incubation of all treated wood samples, the genes coding for oxidative metabolic activity had higher expression levels than the untreated control. In the end of the incubation most of these genes were less expressed than in the untreated control. The genes used for carbohydrate metabolism and the alcohol oxidase showed a significant decrease after 14 weeks of incubation. At the same time an increase in gene expression of an enzyme putative involved in lignin decomposition was detected.
- The use of molecular methods in field trial evaluation can contribute with important additional information to the standard evaluation methods.

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# SUCCESSION OF STAINING FUNGI ON ACETYLATED WOOD AND THE EFFECT OF SELECTED INFLUENCING FACTORS

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#### ABSTRACT

Wood used in outside applications is susceptible to weathering and photo degradation, which often leads to surface discoloration, loss of brightness and surface deterioration. Research has shown that acetylated wood is more resistant against brown rot, white rot and soft rot, and more dimensionally stable than untreated wood. However, acetylated wood seems still to be disfigured by surface moulds and staining fungi. Samples of acetylated Southern Yellow pine at three different treatment levels; low, intermediate and high acetyl content were exposed at two test sites, Ås (Norway) and Bogesund (Sweden) against north and south from September 2010 until March/May 2011. Considerably more precipitation was recorded in Ås in the initial potential fungal growth phase than in Bogesund. As expected, untreated wood had higher mould ratings than acetylated wood. At Ås the tendency was that samples with low acetyl content had lower mould ratings than samples with higher acetyl content. This effect was not found in Bogesund. This may be due to considerably less precipitation in Bogesund compared to Ås. At Ås samples exposed against north tended to have higher mould ratings than panels exposed against south which could be due by less direct sun causing longer time of wetness and more ideal conditions for mould growth.

Key words: acetylation, moulds, Southern Yellow pine, staining fungi.

#### **INTRODUCTION**

Blue stain and mould fungi are often seen upon undesirable elements on painted coated and unpainted wood. Rain, temperature, photo degradation, condensation, high relative humidity and wind degrade the surface of outdoor exposed wooden claddings making these more susceptible to fungal attack (de Meijer 2001, Williams et al. 2000). For colonization and growth of staining and mould fungi on the wooden surface, moisture content in the material and the relative humidity and temperature in the ambient air are the critical factors (Viitanen 1996). Wood modification is defined as a procedure involving the action of a chemical, biological and physical agent resulting in a desired property enhancement during the service life of the modified wood (Hill 2006). Furthermore, modified wood should be non-toxic under service conditions and end of

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life. Modified wood, e.g. furfurylated wood, acetylated wood and thermally modified wood, possess enhanced properties such as better dimensional stability, in addition to enhanced protection against biological attack (Boonstra et al. 1998, Kamdem et al. 2002, Lande et al. 2004a, b, Larsson et al. 2000, Rowell et al. 1985, Sailer et al. 2000, Schneider 1995, Westin et al. 1998, Westin et al. 2002). Acetylation of wood is performed by reacting wood with acetic anhydride. This process results in esterification of the hydroxyl groups in the wood cell wall and formation of acetic acid as a byproduct (Rowell 2005, Rowell et al. 1994). Acetylated wood shows an increase in resistance against wood degrading fungi (Larsson et al. 2000), and a reduction in hygroscopicity of the wood material (Rowell 1991). On the other hand when considering staining fungi, there are indications that acetylated wood do not resist colonisation and growth of staining fungi more than non-acetylated wood (Beckers et al. 1994, Wakeling et al. 1992). Acetylated wood has in some studies been found to be more susceptible to staining and mould fungi (Gobakken and Lebow 2010, Gobakken et al. 2010, Gobakken and Westin 2008) than other comparable wood substrates. The objectives of this study were to set up a pre-trial to 1) investigate how various levels of acetyl content in the wood affect the speed of colonization, 2) study the effect of cardinal direction on the colonization of staining fungi, 3) investigate the effect of location and climatic factors for the onset of growth of staining fungi.

#### MATERIAL AND METHODS

Test specimens were prepared from acetylated Southern Yellow pine sapwood and untreated Southern yellow pine sapwood. The test was preformed according to a modified version of EN 927-3 (2006). Acetylated wood with three different treatment levels; low (level 1), intermediate (level 2) and high (level 3) acetyl content were included in the test. Samples of untreated Southern Yellow pine were used as reference material. Matching samples were put out in Bogesund, Sweden and Ås, Norway, with samples facing both south and north. The samples mounted facing north in Bogesund were installed at 90° angle. All other samples were installed at 45° angle. The panels were put out September 13th 2010 in Ås and September 17th 2010 in Bogesund. At Ås the panels were evaluated visually for mould coverage (rating from 0=no mould growth to 5=heavy mould growth, according to EN 927-3 (2000) and scanned on a flatbed scanner 6 times between September 13<sup>th</sup> 2010 and May 19<sup>th</sup> 2011. In Bogesund the panels were evaluated visually for mould coverage 3 times in the time period between September 17<sup>th</sup> and March 22<sup>nd</sup>. Statistical calculations were done in JMP 9 (SAS Institute Inc 2010). Weather data from a close-by weather station were collected for the period the panels were exposed. The amount of precipitation was very different for the two test sites. In the beginning of the test period (September 13 -October 31) the total precipitation in Ås was 158 mm with a daily mean temperature of 6.5°C. Bogesund had a total of 29 mm precipitation in the same period (September 17 - October 31) and a daily mean temperature of 7°C.

#### RESULTS

At Ås the untreated panels had visible mould growth at the first evaluation (17 days of outdoors exposure), and an increase in mould rating continued until November 2010 when a maximum rating of 5 was reached (Fig. 1ab). Close to no mould growth was

detected on the acetylated panels at the first evaluation, but at the second evaluation (October 15th) mould ratings from 1 to 3 were recorded. Maximum mould ratings (rating 4-5) were recorded in end of April for the acetylated panels.





**Fig. 1b.** Mould ratings for untreated and acetylated panels exposed <u>south</u>, Ås.

Untreated panels were found to have significantly higher mould ratings than acetylated panels. No significant differences were found between the three treatment levels, although the tendency was that the lowest treatment level had lower mould ratings than the two higher levels (Fig. 2a). This tendency was most evident for panels exposed towards south (Fig. 1b). Panels exposed to the north had slightly higher mould ratings than panels exposed to the south (Fig. 2b) although this difference was not statistical significant.





**Fig. 2a.** Variation in mould ratings plotted against treatment level (0=untreated, 1=level 1, 2=level 2, 3=level 3), Ås.

**Fig. 2b.** Variation in mould ratings plotted against cardinal direction (N=north, S=south), Ås.

In Bogesund visible mould growth were detected later than in Ås (Fig.3ab), and the delay in colonization and disfigurement between the two locations were close to 30 days. No mould growth was detected on the acetylated panels at the first evaluation date (October 27<sup>th</sup>), but at the second evaluation (November 10<sup>th</sup>) mould ratings from 0.5 to 2 were recorded. Untreated panels had higher mould ratings than acetylated panels. No clear difference between the three treatment levels were found, although level 2 treated panels exposed against south seems to have slightly higher mould ratings. Acetylated panels exposed to the south had higher mould ratings than panels exposed to the north



which is the opposite of what was found in Ås. In Bogesund panels were exposed to the north at 90° angle.

**Fig. 3a.** Mould ratings for untreated and acetylated panels exposed <u>north</u>, Bogesund.

**Fig. 3b.** Mould ratings for untreated and acetylated panels exposed <u>south</u>, Bogesund.

#### DISCUSSION

The temperature in Ås and Bogesund were similar in the start of the exposure period (second part of September and October 2010). However, Ås had substantial more precipitation than Bogesund during this time period, which was likely the reason for faster colonization and succession of staining fungi on both acetylated and untreated panels at this site. Untreated panels had faster colonization of staining fungi and higher mould rating through the whole test period at both test sites.

At Ås, level 1 panels (acetylated panels with a low treatment level) had lower mould ratings in the beginning of the exposer period than level 2 and level 3 panels. The ester bond linking the acetyl group to the cell wall polymer can be hydrolysed, although studies (Rowell et al 1993, Rowell 2006) has shown that acetylated wood will under normal service conditions have good stability. However, heavy rainfall (ie. incidents of 20-30 mm a day) at a horizontal or moderate angled (ie. 45°) the surface of the panels will be intensively washed, and it can be questioned if the ester bonds then would become more unstable. If assuming that deacetylation of acetylated wood is dependent on the content of the acetyl groups, one can discuss if acetylated wood at a low treatment level will have faster deacetylation than acetylated wood at higher levels. Deacetylation byproducts that may be leached out may give an additional protection against staining fungi during a a certain time after heavy rainfalls. Bardage (Bardage 2011) showed that there is a fungal toxicity associated with high acetic concentration which is supported by several other studies (Paulose et al. 1989, Schillinger and Villarreal 2010). The panels in Bogesund did not experience the same amount and incidents of rainfall, and the tendency of low mould ratings on level 1 panels were not found.

At Ås the panels exposed to the north had higher mould ratings than the panels exposed south. Longer time of wetness (TOW) due to limited direct sunlight can explain this difference. On the other hand, the panels exposed to the north in Bogesund had slightly lower mould rating compared to panels exposed to the south. The panels exposed north

in Bogesund should therefore have received less direct rainfall since they were mounted vertically, and shorter TOW could explain the lower mould ratings.

Samples of the surface from the panels exposed at Ås were harvested at each evaluation date for future identification of the fungal species using DNA-analysis and traditional microscopy to establish the succession of species that causes the discoloration. Further, chemical analysis will be performed to determine the variation in chemical components on the surface of the panels due to treatment level, cardinal direction and related climatic factors. This work was carried out within the frame of the Competence Centre for eco-efficient and innovative wood based materials (Ecobuild). The project group consists primarily of Gry Alfredsen (Norwegian Forest and Landscape Institute), Stig Bardage (SP Trätek) and Lone Ross Gobakken (Norwegian Forest and Landscape Institute).

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# FUNGAL DISFIGUREMENT OF ACETYLATED WOOD

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#### ABSTRACT

Acetylated Southern yellow pine (SYP) panels were exposed at a test field outside Stockholm, Sweden. After 7 months outdoors exposure at 45° facing south and 90° facing north, panels with different acetyl contents became disfigured by fungi. Since panels facing north also became stained by fungi it is believed that UV degradation may have minor importance for the establishment of fungi on the surface of these panels. It is believed that water dynamics may play a more important roll.

Histological studies showed acetylated SYP wood to contain starch, fats and triglycerides and that these extractives have been redistributed to some extent in the wood structure. The presence of such extractives in acetylated SYP wood is the most probable primary source of nutrients for invading fungi.

The content of free acetic acid in acetylated panels may influence the establishment of fungi on panel surfaces exposed outdoors. The effect of acetic acid on fungal growth was studied in laboratory trials. Fungal spores and starved growing mycelia were exposed to acetic acid in solid agar media. Results show toxicity effects of acetic acid on fungi which are related to acid concentration. The limit concentration of acetic acid that totally prevents the growth of *Aureobasidium pullulans* was studied on liquid cultures. Results revealed the limit acid concentration to be in the range of 0.1 %.

Key words: Acetylation, acetic acid, extractives, disfigurement, fungi.

## INTRODUCTION

Southern Yellow Pine (SYP) is the denomination for a group of southern pines that are the principal sources of softwood products in the United States of America. Wood of the various pines is similar in appearance and difficult to separate into species. Commonly SYP is a mixture of Loblolly pine (*Pinus taeda* L.), Longleaf pine (*Pinus palustris* Mill.), Shortleaf pine (*Pinus echinata* Mill.) and Slash pine (*Pinus elliottii* Engelm.). The heartwood of the southern pines is considered moderate to low in resistance to decay. The sapwood has low resistance to decay and blue stain fungi.

Wood modification through acetylation improves the dimensional stability and biological resistance of wood (Rowell 2005). The reaction with acetic anhydride results in esterification of the accessible hydroxyl groups in wood with the formation of acetic acid as a byproduct (Rowell et al. 1994).

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#### $Wood-OH + CH_3-C(=O)-O-C(C=O)-CH_3 \rightarrow Wood-O-C(=O)-CH_3 + CH_3-C(=O)-OH_3 + CH_3-C(O)-OH_3 + CH_3-C(O)-OH_3$

Acetic Anhydride Acetylated Wood Acetic Acid

The increase in resistance to degradation by wood rotting fungi is among the positive effects induced by acetylation (Goldstein et al. 1961, Beckers and Militz 1994, Beckers et al. 1995). Despite this fact it seems that acetylated wood exposed outdoors is susceptible to fungal disfigurement.

In a field trial of various coated unmodified and modified woods aiming at the modeling of the susceptibility of these substrates to fungal disfiguration, it was observed that acetylated pine (Pinus sylvestris) was the most susceptible wood substrate showing very early fungal colonization (Gobakken and Lebow 2010). Moreover, this substrate was considered more susceptible than untreated pine controls, and also received the highest calculated expected probabilities for mould growth. Acetylation of wood seems to promote mould growth on wood exposed outdoors. This finding is also supported by others (Wakeling et al. 1992, Beckers et al. 1994, Edlund 2004). Furthermore, Johansson and Jermer (2010) tested untreated and acetylated pine sapwood (Pinus sylvestris) with high acetyl content inoculated with mould fungi in an environment chamber at 90 % relative humidity and 22°C for 42 days. Untreated pine sapwood got initial fungal growth after 10 days incubation under these conditions and showed heavy fungal growth after 17 days incubation. On the contrary, acetylated pine sapwood only got the first signs of fungal growth after 42 days incubation. Gobakken and Westin (2008) observed that coated acetylated Scots pine exposed outdoors showed severe superficial mould growth after 3.5 years exposure. In this trial the variety of fungal species on the surface of a variety of painted modified wood panels was limited and dominated by A. pullulans.

It has been suggested that a possible source of nutrients for staining fungi colonizing uncoated acetylated wood surfaces would be lignin breakdown products formed at the surface of wood during exposure to solar UV radiation. Schoeman and Dickinson (1997) verified that *Aureobasidium pullulans*, a blue stain fungus considered to be the principal fungal colonist of wood surfaces, is able to live on weathered wood, utilizing products of lignocellulosic photo degradation. This is due to the fungus ability to use lignin breakdown products as a sole carbon and energy source. However, they also observed that different isolates may vary in their ability to grow on lignin breakdown products.

The reason why disfiguring fungi are capable to grow at the surface of acetylated wood is still not well understood.

A field trial was set up to attempt to study the development of surface fungal growth on untreated and acetylated SYP panels. Regular observations on the performance of the panels lead to further laboratory studies.

#### MATERIAL AND METHODS

#### **Field trial**

Untreated and acetylated SYP boards with three levels of acetyl content (low, medium and high) were prepared. Three boards of each level of acetyl content were used. The boards were cut into two panels which were adjusted to the sample size described in the European standard EN 927-3. One panel from each board was exposed at SP Träteks test field in Bogesund outside Stockholm, Sweden, mounted on a rig at 90° facing north, and the other at 45° facing south. The field exposure stated in September 2010. Inspections were conducted on a regular basis. Occurrence and degree of fungal disfigurement was being recorded according to EN ISO 4628-1.

#### Laboratory studies

Histological techniques were used for localization of wood extractives in untreated and acetylated SYP wood: Iodine for localization of starch, Nile Blue for localization of triglycerides, and Osmium tetroxide for localization of unsaturated. Radial sections of both types of wood were prepared with a sledge microtome, stained, and observed using a light microscope.

Spore suspensions and cultures of starving mycelia growing on water-agar of common mould (*Aspergillus niger* and *Penicillium* sp.) and blue stain fungi (including *A. pullulans*) were used to study the effect of acetic acid on fungal growth. Acetic acid was administrated to small filter paper discs which were placed onto solid nutrient media before inoculation with fungal spores by spraying. Incubation was carried out at 25 °C. For tests with starving mycelia, small filter paper discs containing acetic acid were placed directly onto the cultures and the cultures incubated at 25 °C for 20 days.

Series of liquid nutrient media (1.5 % Malt extract) with decreasing concentrations of acetic acid were used to determine the limit concentration of acid which prevents fungal growth. Equal amounts of spore suspension were inoculated into the culture vessels and these were incubated at 25 °C. This study was conducted only on one strain of *A. pullulans* (CBS 621.80).

#### **RESULTS AND DISCUSSION**

#### Field trial

Untreated panels exposed at 45° facing south showed substantial fungal disfigurement (ratings 3 and 4) after 5 weeks exposure. None of the untreated panels exposed at 90° facing north and none of the acetylated panels exposed at the two positions showed signs of fungal disfigurement. After 12 weeks, untreated panels facing south became heavily disfigured (rating 5) and untreated panels facing north started to show fungal growth (ratings 2 to 3). Furthermore, the acetylated panels with low and medium acetyl content facing south started to get visible fungal growth (rating 2), while acetylated panels with high acetyl content still did not become disfigured. Only one panel with low acetyl content among the acetylated panels facing north showed fungal growth (rating

2). After 27 weeks (approx. seven months), all acetylated panels facing south showed fungal growth (ratings 2 to 4.5). Only one acetylated panel with low acetyl content among the acetylated panels facing north still did not show any signs of fungal growth. The other acetylated panels had visible fungal growth but with much less intensity compared to panels facing south. In general, high acetyl content seems to have a delaying effect on the occurrence of surface fungal growth although large variations in degree of fungal disfigurement were observed within acetyl content levels. Panels with low amount of free acetic acid seem to be more susceptible to surface fungal growth, although this effect is not very clear. Panels facing north (no direct UV radiation) also became disfigured by fungi. This indicates that UV radiation may be of minor importance for the occurrence of surface fungal growth on acetylated wood. Although weather conditions in Sweden where quite dry during the first months of exposure, it is believed that water dynamics may play a more important roll. The possible presence of nutrients (wood extractives) at the surface of the panels could also be a contributing factor. A quick histological studied was conducted to elucidate this matter.

Histological staining of radial sections of seasoned untreated SYP wood revealed the presence of starch, triglycerides and fats in the ray cells. The staining of radial sections of acetylated SYP wood (medium acetyl content) revealed that starch is still present in ray cells. However, starch granules were also observed in the fiber lumens and on pit membranes as a consequence of redistribution due to the partial destruction of the ray tissue during acetylation. Histological staining also revealed the presence of triglycerides and fats in acetylated SYP wood. These were seen associated with other extractives outside the rays. Furthermore, these extractives may have been acetylated as well. Ajuong and Birkinshaw (2004) analyzed the extractive fractions from acetylated spruce and larch with FTIR and concluded that extractives become themselves modified in the acetylation process. Furthermore, they also claim that because of their bulk some of these remain within the wood. The redistribution of nutritious wood extractives in acetylated wood may enrich the wood surface with nutrients that could support fungal growth. The presence of these extractives is the most probable primary source of nutrients for fungi colonizing acetylated SYP wood.

The effect of acetic acid on fungal growth was studied in two ways. Malt-agar plates containing a small filter paper disc with acetic acid were inoculated with fungal spore suspensions. After a few days incubation a growth inhibition zone around the paper discs was clearly visible (Fig. 1).



**Fig. 1.** Malt-agar plates with a paper filter disc containing acetic acid and inoculated with fungal spores. Note the growth inhibition zone around the paper disc.

This result indicates that there is fungal toxicity associated with high acetic acid concentration. This effect is widely known among mycologists and has also been reported in scientific works (Paulose et al. 1989, Schillinger & Villarreal 2010).

In order to investigate the effect of acetic acid on mycelial growth, cultures of starving mycelia were exposed to the acid. A concentration gradient of acetic acid was created in the agar medium by means of diffusion. After a few days incubation clear effects could be seen on the fungal cultures (Fig. 2). A zone of complete growth inactivity was created around the paper discs containing acetic acid. At that zone it is believed that the acid concentration is too high to allow fungal growth. When the concentration of acetic acid decreases the fungal mycelia reacted with the production of pigment and spores. Further away, were the acid concentration should be much lower, the fungal mycelia seemed unaffected. These results indicate once more that there is fungal toxicity associated with high acetic acid concentration and that at certain acid concentrations fungi seems to be put on stress with increased metabolic activity.



**Fig. 2.** *A. pullulans* starving mycelium exposed to acetic acid. Acetic acid was administrated to the filter paper disc on the left. The paper filter disc on the right, without acid, acts as control.

To verify what would be the limit acetic acid concentration that allows the growth of *A*. *pullulans*, a series of laboratory trials using liquid cultures with decreasing concentrations of the acid were conducted. An acetic acid concentration of 0.1% or higher inhibited totally the germination and growth of *A*. *pullulans* spores. Spores started to germinate and produce mycelia at an acid concentration of 0.05%. Furthermore, even here the level of metabolic activity seems to be higher with more mycelial production compared to a control culture without acetic acid.

The present work was carried out within the frame of the Competence Centre for ecoefficient and innovative wood based materials (Ecobuild, http://www.ecobuild.se). The project group is primarily composed by Stig Bardage (SP Trätek), Lone Ross Gobakken and Gry Alfredsen (Norwegian Forest and Landscape Institute).

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# MOULD GROWTH RESISTANCE OF FUNGICIDE-CONTAINING WPCs

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#### ABSTRACT

Extruded wood plastic composites (WPCs) are nowadays an established building material in Europe. The advantages of WPCs compared to solid wood for outdoor products are for example dimensional stability and less maintenance, mainly due to a lower moisture sorption rate in the composites. One feature of WPCs shared with solid wood products is the risk of mould growth on the surface of the material. In this study, a commercial extruded WPC profile with and without added fungicide has been evaluated with respect to resistance to mould growth. The experiment was performed in an environmental chamber, according to ASTM D3273. The results indicate a significantly lower mould growth rate on all the WPCs formulations compared to solid wood coated with a distemper paint as control. No mould growth was observed after four weeks test on one type of fungicide-containing WPC.

Key words: WPC, mould, fungicide.

#### INTRODUCTION

Wood plastic composites (WPCs) are composed of wood in the form of particles or fibres, a thermoplastic matrix partly encapsulating the wood component and additives. The moisture sorption in such composites is several magnitudes slower than for solid wood. However, there may be a large gradient in moisture content of WPCs and the moisture content in their surface regions can reach sufficient high levels for fungal growth (Ibach et al. 2011, Gnatowski 2009, Wang and Morrell 2004). Therefore, in outdoor use, there is a need to protect WPC from surface mould growth. One conventional mean to avoid fungal attack in WPCs is to incorporate zinc borate into the formulations. Zinc borate is effective against wood decay fungi and insects but not highly effective against mould and staining fungi (Shirp et al. 2008). Dylingowski (2003) has shown that a fungicide system containing dichloro-octyl-isothiazolone (DCOIT) incorporated in WPC is more effective against mould and staining fungi than zinc borate. Johansson and Jermer (2010) performed a comparative study on the mould

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growth on different wood based materials, there an injection moulded WPC with isothiazolone based fungicide performed well against mould growth.

Mould is a group of non-degrading fungi, which usually feeds on nutrients present in wood leaving cellulose, hemicelluloses and lignin intact. Mould fungi grow rapidly on any surface in the presence of low molecular nutrients and moisture producing great amounts of spores. Mould fungi may cause health problems such as allergies or create problems for those already have pulmonary conditions (Klyosov 2007). Furthermore, the discoloration of certain building materials caused by mould fungi is often not acceptable by the end user due to aesthetical reasons.

The objective of this study was to evaluate the mould growth resistance of a commercial wood plastic composite with and without the use of metal free preservatives.

#### MATERIALS AND METHODS

#### Materials

The following three wood plastic composite (WPC) decking boards with a cross section of 145 x 25 mm<sup>2</sup> were prepared at OFK Plast AB, Karlskoga, SWEDEN: 1) a reference WPC with the standard BeachPlank® recipe, 2) WPC with the BeachPlank® recipe with addition of 800ppm DCOIT in dry wood component using a DCOIT based formulation; and 3) WPC with the BeachPlank® recipe with addition of 0.5% BKC in dry wood component using a BKC based formulation. The DCOIT and BKC based fungicides were supplied by Viance LLC, Charlotte, NC. The upper face of the boards had a brushed surface and the lower side had a surface as created when exiting the extruder die.

#### Test method

The set up for testing the resistance of growth of mould were according to ASTM D3273-94. Specimens were suspended ca 50 mm above mould-fungi containing soil inside a small environmental chamber (see Fig. 1). The chamber had a temperature of 27 °C and 98-100% relative humidity. The test duration was 4 weeks and the specimens were rated after 1, 2, 3 and 4 weeks regarding the degree of fungal coverage on the surfaces. The rating was between 0 (no growth) and 4 (60% to complete microbial coverage). Ratings 1, 2 and 3 represents 0-10%, 10-30% and 30-60% fungal coverage respectively.

Specimens measuring  $120 \times 50 \times 7 \text{ mm}^3$  were prepared from both the upper and lower face of the boards and were placed in 23 °C, 65 % relative humidity room for one month prior to the mould test. Figure 2 shows the sawing pattern. 6 specimens of each formulation and side were prepared out of which 5 specimens were used in the mould test and one was saved for reference, see Table 1 for materials and specimens. Control panels with known susceptibility to mould growth were prepared from Scots pine coated with a red laboratory distemper paint without fungicides.



Fig. 1. Specimens suspended in the environmental chamber.



Fig. 2. Schematic drawing of specimen preparation from the WPC boards.

Table 1. WI C matchais tested .							
	Europiaida	Fungicide	Fungicide Number of specimens				
	rungicide	content	Upper side	Lower side			
BeachPlank®	-	-	6	6			
SP1	DCOIT	800ppm	6	6			
SP2	BKC	0.5%	6	6			
#### **RESULTS AND DISCUSSION**

The results from the ASTM D3273-94 mould growth test showed that the control specimens were heavily covered with a thick layer of mould already after one week exposure, and that all of the control specimens were completely covered with mould after 2 weeks. This indicates that the fungal activity in the chamber was good. The fungal growth on the WPC samples showed a much more sparse coverage than for the controls, if any growth at all. This could be attributed to the less amount of accessible nutrition for the test fungi on the WPC surfaces. Fig. 3 shows the rating after each week of the test. The brushed upper side of the BeachPlank® showed the most rapid growth of the WPC materials. After 4 weeks, the mould growth on the BeachPlank® on both sides was similar to the growth on the SP2 WPC on both sides, indicating that the BKC based formulation did not have an effect on resistance to mould growth. The SP1 WPC performed excellent with no growth after the test period. Fig. 4 displays photographs of specimens after 4 weeks exposure. It can be noted that the Ref 1:4 and the SP2 13:4 specimens were rated 3, and compared to the distemper paint controls, these specimens showed a much less thick coverage. However, the rating was determined on the area covered and not the intensity.



Fig. 3. Rating of the samples in the environmental chamber.



**Fig. 4.** Specimens after mould chamber for 4 weeks, from left: Reference WPC, SP1 WPC, SP2 WPC, and limewash control.

#### CONCLUSIONS

The mould growth on the surface of WPCs was in general much less compared to the reference material. This is primarily due to less content of available nutritious substances at the surface of WPCs. Mould growth on this material was entirely avoided by using 800ppm DCOIT in the composite. The BKC based formulation had no effect on the mould growth at 0.5% of BKC loading.

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# CHANGES IN THE CHEMICAL AND MECHANICAL PROPERTIES OF ASH-TREE WOOD AFTER HYDROTHERMAL MODIFICATION

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## ABSTRACT

Ash-tree wood is heavy, tough and durable; hence, it belongs to hard deciduous wood species. In comparison with oak, ash-tree is more fast-growing; it reaches a height of 20 m in mixed forests. Ash-tree wood is used for producing joinery articles and furniture. It has very good mechanical properties and can be very easily joined with nails, bolts and glue. However, ash-tree wood has a low durability against rot, wood borers and different microorganisms; therefore, to extend its applicability in outdoor conditions, it is necessary to improve the biodurability properties of wood. In the hydrothermal modification process, not only the chemical properties and structure of wood are changed, but also physical properties such as colour, mass, volume and mechanical strength. The heating of wood in the water vapour medium influences mainly the three basic components of wood - cellulose, hemicelluloses and lignin.

In the present study, hydrothermal modification of ash-tree wood was carried out in an experimental laboratory plant in water vapour medium. Ash-tree boards were modified at 4 different temperatures - 140, 160, 170 and 180°C. Mass and density changes in the thermal treatment were determined. With increasing treatment temperature, density decreases, but mass losses grow, which can be explained by the evaporation of more volatile components. To study the chemical composition, extraction with acetone, cellulose determination according to the Kürschner-Hoffer method, lignin determination according to the Klason method and holocellulose determination according to the peroxyacetic acid method were performed. After the Klason lignin isolation from wood, a filtrate sample with dissolved sugars was taken, and analysis was carried out using a liquid chromatograph to determine the amount of monosaccharides in wood. This gives the idea of the hemicelluloses destruction in thermal treatment. The changes in the chemical composition (destruction of hemicelluloses) inevitably lead to the worsening of mechanical properties; therefore, to control these properties, bending strength measurements and determination of Brinell hardness were carried out.

Key words: ash tree, mechanical properties, chemical composition, hydrothermal modification

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# INTRODUCTION

Recently, for economic and ecological reasons, the tendency of wider use of the wood of local tree species grows. This is determined by several factors, namely, the accessibility of high-quality and tropical wood decreases, and the demand for renewable, environmentally-friendly materials for indoor and outdoor applications grows.

The total stock of deciduous trees in the Republic of Latvia is  $\sim 251$  million m<sup>3</sup>, from which approximately a half (56%) is birch, 22% is aspen, 20% is alder, and 2% is ashtree. The natural durability of these tree species against the biological action, UV irradiation, humidity and other factors is limited. Worldwide, the modification of soft deciduous wood in a water vapour medium at elevated pressure is poorly investigated. In the recent years, studies have been initiated in Latvia on the use of soft deciduous wood for producing products with a higher added value, but practically, there are no studies on the thermal modification of hard deciduous wood (ash-tree, oak). As optimum regimes for thermal modification of deciduous wood (aspen, birch, ash-tree), temperatures from 180 to 220°C are recommended. In this temperature range, the mechanical properties of wood as a material are changed dramatically. Therefore, to forecast the properties of the modified material as well as its stability in service conditions, it is important to elucidate not only the properties of the obtained product, but also to understand the processes occurring in wood as a result of the thermal action, carrying out studies on the changes in the chemical composition of wood, as well as microstructure, physical and mechanical properties.

# MATERIAL AND METHODS

Ash-tree boards without any visible defects were chosen for modification. Samples sizes were: length  $1000 \pm 2$  mm, width  $100 \pm 0.5$  mm, thickness  $40 \pm 0.5$  mm. The average density of ash-tree wood at the absolute moisture content 12% was 784 kg/m<sup>3</sup>. Absolute moisture content of the wood before modification was 5-7%.

The thermal modification was carried out in a multifunctional wood modification pilot device (WTT). Ash-tree boards were thermally modified in the water vapour medium for 1 h at 4 different temperatures: 140°C, 160°C, 170°C and 180°C and for 3 h at 160°C. The material was placed in an autoclave, in which 0.45 ml of water was supplied per 1 g of oven dry wood.

**Chemical analyses** were performed for both untreated wood and the modified samples. 8-12 g of air dry chips of each sample were extracted in a Soxhlet apparatus with acetone for 8-10 h for extractives determination. Cellulose was determined applying the Kürchner-Hoffer method; for lignin determination, the Klason 72% sulphuric acid method was applied. For holocellulose content determination, 10% peracetic acid was used (Browning 1967).

After Klason lignin isolation, filtrate with the dissolved monosaccharides was taken. The filtrate was neutralized till pH 5-7. Chromatography analysis using Shimadzu HPLC was carried out. For obtaining a calibration plot, standard solutions (ramnose, xylose, arabinose, mannose, glucose and galactose) of different concentration were prepared. The obtained results were recalculated in % from the mass of the oven dry weighed amount before the lignin isolation.

**Bending strength.** For each treatment series, 25 parallel samples with the sizes 20 x 20 x 360 mm without visible faults were chosen, with fibers parallel to the longitudinal direction. The difference of densities for parallel samples was in the range of  $\pm$  10%. Samples were placed in a condensing chamber at a temperature of  $20 \pm 2^{\circ}C$  and the relative humidity of air 65  $\pm$  2% until the constant mass was reached. Wood properties in bending were determined according to the requirements of the DIN 52186 (1978) standard. Bending strength of wood was determined, using a material strength testing device ZWICK Z100. Speeds of loading were appropriate for each group individually, so that to achieve the destruction maximum within 90  $\pm$  10 sec.

Wood surface hardness according to Brinell. For untreated and thermally treated ashtree wood, hardness was determined by the LVS EN 1534 test method. Using the material hardness testing device, the ball semisphere (ball diameter 10 mm) was pressed into the wood with a constant strength of 1000 N. The semisphere impress hollow diameter was determined with the help of a reflecting light binocular microscope. Sample sizes were:  $25 \times 25 \times 50$  mm without visible faults, parallel to the annual ring direction. Those were conditioned for 4 weeks at a temperature of  $20 \pm 2^{\circ}C$  and the relative humidity of air  $65 \pm 2\%$ . The difference of densities for parallel samples was in the range of  $\pm 10\%$ . For each series, 20 parallel samples were chosen.

Treatment temperature/time	Mass loss (%)	Density ( $W_{rel} = 12\%$ ) (kg/m <sup>3</sup> )	Density loss ( $W_{rel} = 12\%$ ) (%)
Untreated	-	784	-
140/1	5.4	756	3.6
160/1	7.3	741	5.5
160/3	9.3	721	8.0
170/1	16.5	664	15.3
180/1	17.7	657	16.2

## **RESULTS AND DISCUSSION**

**Table 1.** Changes in the physical parameters of ash-tree wood depending on the modification temperature.

With increasing treatment temperature, density for ash-tree decreases, and mass losses grow (Table 1). Relative mass losses embrace: water evaporation, evaporation of extractives, evaporation of the products of destruction of wood components, especially products of hemicelluloses destruction (Kocaefe et al. 2008).

With increasing treatment temperature, the amounts of acetone soluble extractives grow 5-10 times (Table 2.). The increase of extractives can be applied to hemicelluloses destruction into easier volatile compounds, which are then obtained in extraction (Manninen et al. 2002). With increasing temperature, relative amounts of cellulose and lignin in ash-tree wood also grow. It is observed that, varying the treatment parameters (temperature, holding time, pressure), the relative amount of crystalline cellulose grows

(Bhuiyan and Hirai 2000). However, it is not known whether this growth is connected with the destruction of the amorphous region, or also crystallization processes, or both the processes simultaneously. It can be concluded that, during the hydrothermal treatment, macromolecules are partially governed by thermal destruction, the extent of which is determined by the degree of macromolecules crystallinity. With decreasing polysaccharides and increasing mass losses, as a result of the thermal treatment, the relative content of lignin grows.

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Treatment temperature/ time	Acetone soluble extractives (%)	Cellulose (%)	Lignin (%)	Holocellulose (%)	Hemicelluloses (%)	Hemicelluloses * (%)
Untreated	1.4	49.6	24.9	68.2	25.5	18.6
140/1	1.1	49.9	26.3	62.8	23.8	12.9
160/1	7.7	52.5	30.2	60.2	17.3	7.7
160/3	10.1	56.7	30.7	62.5	12.6	5.8
170/1	13.1	60.7	31.9	65	7.4	4.3
180/1	14.9	61.3	36.8	62.5	1.9	1.2
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**Table 2.** Changes in the chemical composition of ash-tree wood depending on the modification temperature.

Hemicelluloses = 100 – (Cellulose + Lignin) Hemicelluloses \* = Holocellulose - Cellulose

With increasing treatment temperature, no regularity is observed for changes in the holocellulose amount. The calculated yield of hemicelluloses<sup>\*</sup> is lower than that calculating hemicelluloses = 100 - (Cellulose + Lignin), but the tendency is similar. This makes to conclude that these isolation methods are difficult to compare, and cellulose would have to be isolated from holocelluloses, so that to determine the definite content of hemicelluloses.

**Table 3.** Monosaccharides in the filtrate after the distribution of Klason lignin.

Treatment temperature/ time	Xylose (%)	Glucose (%)
Untreated	25.9	40.6
140/1	23.9	41.8
160/1	23.5	40.8
160/3	21.2	46.4
170/1	15.4	51.5
180/1	10.4	57.6

With increasing treatment temperature, in the filtrate, after isolating Klason lignin, the relative amount of glucose grows, but the relative amount of xylose considerably (2.5 times) decreases (Table 3.). Thermally modifying wood, the amounts of xyloses, arabinoses, galactoses and mannoses decline (Esteves et al. 2008). Xylose, which is present in the hemicellulose molecule, obeys thermal destruction most readily. At temperatures above 230°C, the amounts of xylose and mannose in wood decrease, but arabinose and galactose disappear at all, converting into thermolysis end products (Zaman et al. 2000). The growth of the glucose amount is connected with the relative

increase in the cellulose amount (glucose is the basis of the cellulose structure). Because xylose is the basis of the thermally unstable structure of hemicelluloses, its amount decreases. Other monosaccharides were not detected by our HPLC equipment.

Treatment	Hardness according to Brinell				
time	Tangential surface	Radial surface			
Untreated	$3.50\pm0.27$	$3.20\pm0.15$			
140/1	$3.27\pm0.13$	$3.45\pm0.08$			
160/1	$2.70\pm0.16$	$2.45\pm0.10$			
160/3	$2.33\pm0.16$	$2.32\pm0.11$			
170/1	$2.64\pm0.15$	$2.11\pm0.09$			
180/1	$2.21\pm0.09$	$1.97\pm0.07$			

Table 4. Hardness according to the Brinell EN LVS 1534 test method.

With increasing treatment intensity, both tangential (except 170°C) and radial (except 140°C) surface hardness for ash-tree decreases (Table 4.). It is mentioned that thermal modification increases the wood hardness, but also the opposite effect is recorded, namely, the wood becomes softer (Poncsak et al. 2006, Gunduz et al. 2009). It can be concluded that the tangential surface hardness is by 5-20% greater than that for radial surface, which is probably explained by the densification of the structure in the radial direction. The decrease in hardness is caused by the decrease in density, which develops mainly due to the destruction of hemicelluloses.

Table :	Table 5. Bending strength and modulus of elasticity.								
Treatment temperature / time	Modulus of elasticity (N/mm <sup>2</sup> )	Bending strength (N/mm <sup>2</sup> )	Decrease of bending strength compared to control (%)						
Untreated	$11475 \pm 1138$	$111 \pm 17$	0						
140/1	$11786\pm1192$	$110\pm19$	1						
160/1	$13120\pm1297$	$97\pm23$	13						
160/3	$12575\pm1315$	$90\pm26$	19						
170/1	$11925\pm1255$	$86\pm23$	23						
180/1	$11541 \pm 1249$	$79\pm21$	29						

 Table 5. Bending strength and modulus of elasticity

Modulus of elasticity for ash-tree grows at the first treatment regimes (140°C and 160°C/1h), then decreases (Table 5). However, the same tendency was obtained by Kobujima et al. (2000). With increasing treatment temperature, bending strength decreases. Bending strength for unmodified wood and that modified at 140°C differs little. At 180°C, bending strength losses for ash-tree, in comparison with the case of the initial wood, reach 29%, which is a good indicator, because even up to 50% strength losses are reached for pine and eucalyptus at such temperatures (Esteves et al. 2007).

The decrease in strength is explained by the thermal destruction of hemicelluloses, mainly xylan (which is testified mainly by the analysis of monosaccharides), which is the basic substance for cellulose and lignin.

# CONCLUSIONS

With increasing hydrothermal treatment temperature for ash-tree wood:

- 1. Density decreases and mass losses grow;
- 2. Relative amounts of extractives, cellulose and lignin grow. No linear relationship is observed for the changes in the holocellulose amounts. The calculation testifies that the amount of hemicelluloses decreases.
- 3. In the filtrate, after the isolation of Klason lignin, the relative glucose amount increases, and the relative amount of xylose decreases.
- 4. Surface hardness, both in tangential and radial direction, decreases. Tangential surface hardness is higher by 5-20% than that for the radial surface.
- 5. Modulus of elasticity grows at the first treatment regimes  $(140^{\circ}C \text{ and } 160^{\circ}C/1h)$ , then decreases. Bending strength decreases.

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# CHEMICAL CHANGES OF UNTREATED AND HYDROTHERMALLY MODIFIED HARDWOOD AFTER ARTIFICIAL WEATHERING

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#### ABSTRACT

Outdoors, wood degrades due to weathering from factors such as solar radiation (ultraviolet, visible and infrared light), water (rain, dew, snow, humidity), temperature and atmospheric pollution (sulphur dioxide, nitrogen dioxide, ozone, dust, etc.). Among these factors, solar radiation, especially ultraviolet light, has the most damaging effect by the formation of free radicals and depolymerizing lignin in the wood cell walls, followed by water, which leaches the degradation products, causing surface erosion.

This study examined three species of soft deciduous wood – aspen (*Populus tremula*), birch (*Betula pendula*) and grey alder (*Alnus incana*), all of which were weathered untreated and hydrothermally modified (HTM-d) at 160°C for 3 h and at 170°C for 1 h. The artificial weathering period was 1000 h and consisted of cycles of condensation, irradiation with UV light and water spray. Because the photodegradation of wood is essentially a surface phenomenon, the ATR FTIR method was applied to examine the changes in the chemical structure of wood. The spectra showed that the characteristic peaks of lignin in all examined wood species (untreated and after hydrothermal modification (HTM) significantly decreased or practically disappeared after weathering, which means that lignin becomes most degraded. Cellulose, which consists of amorphous and crystalline regions, is also affected by weathering, and the amorphous part of cellulose is more degraded. The results show that the HTM-d wood surface after weathering also becomes damaged and needs to be protected; therefore, the next aim of the work will be to find coatings with the best protection properties.

Key words: HTM-d wood, artificial weathering, ATR FTIR.

## **INTRODUCTION**

Wood is a naturally durable material that has been recognized for centuries worldwide for its versatile and attractive engineering and structural properties. When wood is exposed outdoors above the ground, a complex combination of chemical and mechanical factors contributes to what is described as weathering – a complex set of reactions induced by the following factors: solar irradiation (ultraviolet, visible and infrared light), water (rain, dew, snow, humidity), temperature and atmospheric pollution (sulphur dioxide, nitrogen dioxide, ozone, dust, etc.) (Anderson 1991).

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Exposure of wood to UV light is known to be mainly responsible for the degradation and discoloration of the wood surface in weathering (Budakci 2006).

Artificial weathering is used to elucidate the chemical changes at the wood surface after weathering, although the results are not the same as those after natural weathering. The weathering process of wood is primarily a surface phenomenon, although the cracks developing during weathering can be sensitive to the fungal attack and lead to a more severe destruction of wood. This research was carried out to gain more information about hydrothermally modified (HTM-d) soft deciduous wood weathering processes, which is very important for using wood outdoors.

Artificial weathering usually consists of cycles of UV irradiation and water spray. In this research, this method was applied to establish how the hydrothermal modification (HTM) changes the chemical processes during weathering. Irradiation with only UV light was used to find out how exactly UV light affects soft deciduous wood species.

# MATERIAL AND METHODS

For the research, three species of soft deciduous wood were used, namely, aspen, birch and grey alder. Untreated samples of all species and those HTM-d at  $160^{\circ}$ C for 3 h and at  $170^{\circ}$ C for 1 h were artificially weathered. These two regimes for HTM were chosen as a potential optimum for soft deciduous wood to improve durability. Two types of artificial weathering were used, namely, weathering for 1000 h, which consisted of cycles of condensation, irradiation with UV light and water spray, and artificial weathering for 260 h with UV irradiation alone. The samples used in artificial weathering, including water spray and UV irradiation, were taken only after a full period, but the samples used for artificial weathering with UV irradiation were taken after 2, 10, 35, 100 and 260 h.

From all specimens, surfaces were cut off (dimensions – 0.6 cm x 0.6 cm x 0.1 cm) and vacuum dried and, as the photodegradation of wood is essentially a surface phenomenon, the ATR FTIR method was applied to examine the changes in the chemical structure of wood (Muller 2002). Spectra were measured at a resolution of 4 cm<sup>-1</sup> and 64 scans were recorded per sample in the range of 4000–600 cm<sup>-1</sup> using a Perkin-Elmer Spectrum One with Perkin Elmer Universal ATR Sampling Accessory.

# **RESULTS AND DISCUSSION**

In ATR FTIR spectra, the most characteristic region of chemical changes after HTM and artificial weathering is from  $1750 \text{ to } 850 \text{ cm}^{-1}$ .



**Fig. 1.** ATR FTIR spectra of aspen wood in the region 1750-850 cm<sup>-1</sup>.

It can be concluded from the spectra (Fig. 1) that the bands peculiar to lignin at 1600, 1510 cm<sup>-1</sup>, after weathering, disappear or their intensities decrease, and also after HTM, they become wider and slightly shift leftwards. The intensity of the peak at 1465 cm<sup>-1</sup> (characteristic of lignin) after HTM increases, and after weathering, it practically disappears. The peak at 1730 cm<sup>-1</sup>, characteristic of hemicelluloses and lignin after modification, is much smaller and wider; after weathering, it has practically disappeared. Among the wood constituting polymers, lignin is the most sensitive one to light. Photochemical reactions are initiated by the absorption of UV-visible light mainly by lignin, which leads to the formation of aromatic and other free radicals. These free radicals may then cause the degradation of lignin and photo-oxidation of cellulose, and unsaturated carbonyl compounds resulting in colour changes and yellowing of the wood surface (Pandey 2005).

The peaks characteristic of cellulose, for example, at 1315 cm<sup>-1</sup> for untreated aspen, are wide and with a "shoulder"; after modification it gets higher and, after weathering, appears as a new small peak at 1333 cm<sup>-1</sup>. These changes are explained as an increase of the rate of crystalline cellulose and, at the same time, a decrease of the rate of amorphous cellulose (Colom 2003). The peak at 1423 cm<sup>-1</sup> is peculiar to amorphous and crystalline cellulose but, as can be seen, after modification, this peak slightly shifts leftwards and, after weathering, it is at 1430 cm<sup>-1</sup>, which is characteristic of crystalline cellulose. The peak at 1156 cm<sup>-1</sup> belongs to amorphous cellulose and, after HTM and weathering, it gets higher and slightly shifts leftwards. In the literature, it is described that the peak at 1156 cm<sup>-1</sup> is more characteristic of amorphous cellulose and at 1163 cm<sup>-1</sup> of crystalline cellulose (Colom 2003). The peak at 1100 cm<sup>-1</sup>, which is peculiar to cellulose after HTM and weathering, gets higher.

The peak at 1030  $\text{cm}^{-1}$  for untreated aspen is very wide and high, it also has a "shoulder" in the left side and, after HTM and weathering, it appears as a small peak, which is defined as the peak of degradation products. The peak at 895  $\text{cm}^{-1}$  is

characteristic of cellulose and hemicelluloses and no specific changes are observed there.



**Fig. 2.** ATR FTIR spectra of birch wood in the region 1750-850 cm<sup>-1</sup>.

If the spectra of all three species are compared, namely, aspen, birch and grey alder (Figs. 1-3), then it can be seen that they all are quite similar and there are no typical differences between these soft deciduous wood species.



**Fig. 3.** ATR FTIR spectra of grey alder wood in the region 1750-850 cm<sup>-1</sup>.

Because ultraviolet light has the most damaging effect on wood, it is useful to compare the changes between the full weathering and weathering only with UV irradiation. Fig. 4 shows the chemical changes of grey alder depending on time for weathering with UV irradiation, and it can be seen that this is a gradual process and depends on the time of UV irradiation.



**Fig. 4.** ATR FTIR spectra of grey alder wood after weathering with UV irradiation in the region  $1750-850 \text{ cm}^{-1}$ .

The peaks characteristic of lignin at 1600 cm<sup>-1</sup>, 1510 cm<sup>-1</sup> and 1465 cm<sup>-1</sup> after weathering with UV light become smaller and wider, slightly shift leftwards, and after 260 h, practically disappear.

The peak at 1423 cm<sup>-1</sup> is peculiar to amorphous and crystalline cellulose but, as can be seen, after weathering with UV irradiation, this peak gets wider and somewhat shifts leftwards to 1430 cm<sup>-1</sup>, which is characteristic of crystalline cellulose. The peak at 1156 cm<sup>-1</sup> belongs to amorphous cellulose and, after weathering with UV irradiation, gets higher and slightly shifts leftwards. The peak at 1100 cm<sup>-1</sup>, which is peculiar to cellulose after weathering with UV light, does not change.

The peak at 1030 cm<sup>-1</sup> for untreated grey alder is very wide and with a high intensity, it also has a "shoulder" in the left side and, after weathering with UV irradiation, becomes somewhat higher. The peak at 895 cm<sup>-1</sup> is characteristic of cellulose and hemicelluloses, and no specific changes are observed there.

The greatest changes, comparing the chemical changes between the full weathering and the weathering with UV irradiation, are at 1233 cm<sup>-1</sup> and 1730 cm<sup>-1</sup>. The peak at 1233 cm<sup>-1</sup> describes C-C, C-O and C=O stretches. After the full weathering, this wide band disappears, but after the weathering with UV irradiation, it is the same as for untreated wood. The peak at 1730 cm<sup>-1</sup> is characteristic of hemicelluloses and lignin and, as can be seen, after weathering only with UV irradiation, gets higher, which is opposite to that after the full weathering.

Those typical chemical changes were observed in all three species of soft deciduous wood, namely, grey alder, aspen and birch.

# CONCLUSIONS

As a result of artificial weathering (water spray + UV irradiation) of both untreated and HTM-d wood species, all three soft deciduous wood species mostly degrade lignin and amorphous cellulose, and the surface of wood becomes damaged.

Artificial weathering with UV irradiation is a gradual process, and the chemical changes after the full weathering and the weathering with UV irradiation of soft deciduous wood are different.

Soft deciduous wood after HTM needs protection from degradation processes with special coatings.

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# THERMAL MODIFICATION OF BIRCH USING SATURATED AND SUPERHEATED STEAM

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#### ABSTRACT

During the thermal modification, wood is normally exposed to temperatures between 160 - 220°C. As a result physical and chemical changes are taking place and some of the wood properties are changed. Dimensional stability and weather resistance are improved. On the other hand the mechanical strength properties are usually negatively affected by the treatment. The visual appearance is also changed. There were two different types of thermal modification processes used in this study. One of them was using saturated steam and the other one superheated steam. Treatment temperature was 160°C in saturated steam process and 185°C in superheated steam. The wood specie used in this study was Silver birch (*Betula pendula*). In the chemistry part the acid content was investigated. Despite the 25°C lower treatment temperature, birch modified in saturated steam was more acidic compared to birch modified in superheated steam. Some differences in equilibrium moisture content (EMC) and dimensional stability were found mainly in the environment T=20°C and RH=85%. The colour of birch treated in superheated steam at temperature 185°C.

Key words: Saturated steam, Superheated steam, Thermal modification, Heat treatment

#### INTRODUCTION

Environmental aspects have increased interest to develop new, alternative wood modification methods. Thermal modification using adjustable treatment parameters is one of them. Thermal modification of wood causes both chemical and physical changes. The changes depend on different factors such as process, treatment parameters, wood species, moisture content and sometimes even on dimension of treated wood. Usually the thinner, having smaller cross-section samples are easier to treat. The major part of property changes during the treatment process are caused by chemical reactions taking place in the wood cell structure. It is well known that hemicelluloses are degrading during thermal modification process (Stamm 1956, Kollman and Fengel 1965, Tjeerdsma et al 1998, Sivonen et al. 2002). Acetyl groups of hemicelluloses are split of during heating and as a result acetic acid is formed (Kollman and Fengel 1965). In hardwoods, acetic acid formation is mainly due to the degradation of the hemicellulose glucuronoxylan (Theander and Nelson 1998). Low- molecular organic aids such as acetic and, especially, formic acid are volatile and are difficult to collect and analyse (Sundqvist et al. 2006). Dimensional stability of heat treated wood is related to

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degradation of polysaccharides and hemicelluloses as they can bond water in cell wall and are greatly influenced by the process. This degradation process leads to formation of products that interact less with water but also to evaporation and leaching during the heat treatment. It is well known that cellulose in wood contributes considerably to its mechanical properties. Sundqvist et al. (2006) found that treatments for birch at 180°C for 1 to 2.5 hours reduced strength and hardness significantly. Losses in mechanical properties can be linked to the mass loss and increase in formic and acetic acid concentrations. Time and especially the heat-treatment temperature have a significant influence on changes in colour. It has been shown that hygrothermally treated birch has a rapid decrease in lightness early in the heat treatment process and at fairly low temperature between 160°C and 180°C

#### MATERIAL AND METHODS

#### **Raw material and processes**

Wood specie used in this study was Silver Birch (Betula pendula) from Eastern part of Finland. Birch was through and through sawn and seasonally dried and then further kiln dried in vacuum to moisture content approximately 12%. The drying temperature was kept fairly low in order to avoid any type of discoloration. After kiln drying the 4 - 4.5meter long and 32 mm thick boards were cut into three parts. One part was used as a reference, second and third parts were thermally modified in superheated and saturated steam respectively. Number of boards in each batch was 20. Density of untreated wood was 543 kg/m<sup>3</sup> as an absolute dry. Superheated modification was carried out in a typical kiln drying chamber. The process is commonly known as a Thermowood® process. The process is using steam as a heat transfer medium and an inert blanket to limit oxidative processes. Normally in Thermowood process the treatment temperatures are varying between 180°C-215°C. Treatment temperature used in this study was 185°C having two hours duration in maximum temperature. These treatment parameters meet the requirements set for class Thermo-S. The process using saturated steam is carried out in a pressurized (5-8 bar) autoclave cylinder while the process using superheated steam is operating all the time at normal atmosphere in a typical drying chamber. There is not any particular, intensive drying phase during the process. Process is semi-closed having a possibility to release pressure if necessary. Treatment temperature used in this process was 160°C having one hour duration in maximum temperature.

#### **Test methods**

#### Colour measurement

The colour of wood was measured from planed and defect free surface. There were three measurement points in each sample, taken into consideration the whole width of the board. The shown value is the average from these measurements. The colour measurement device used was Minolta Chroma Meter CR210. It measures the colour in a three dimensional colour space according to CIE L\*a\*b\* system and standard. L\*a\*b\* colour space uses rectangular coordinates. In this colour space, L\* indicates lightness varying between L\*= 0 (black)...L\*=100 (white), a\* and b\* are the chromaticity coordinates. In chromaticity diagram a\* and b\* indicate colour directions: +a\* (red) direction, -a\* (green) direction, +b\* (yellow) direction, and -b\* (blue) direction.

#### *Equilibrium moisture content (EMC)*

EMC was determined in conditioning chamber with constant  $20^{\circ}$ C temperature and three different relative humidity (RH) settings; RH 35 %, / RH 65 % / RH 85 %. Size of the samples was 200 (l) x 100 (w) x 20 (t) mm. All the tested batches had 22 samples. The final moisture content was determined by using oven dry method.

#### Dimensional stability

Dimensional stability was determined in same conditions as EMC tests. Dimensions of the samples were measured in longitudinal, tangential and radial direction. The results are expressed as a swelling percentage (%) and then compared to absolute dry (0%) dimensions.

#### Acidity (pH)

The samples for the tests were milled from defect free boards. The saw dust (1g) was mixed with 20 ml water solution with 3% NaCl and put in ultrasonic bath for 120 minutes. The pH was measured with pH meter Metrohm 744. Titration of acid equivalents in the solutions was performed with sodium hydroxide (NaOH) 0.01002M. Measurements were repeated after 24 and 96 hours before the solutions were finally neutralized.

#### RESULTS

#### **Colour measurements** (L\*a\*b\*)

The colour measurements showed that both the treatments result in significant L\*-value decrease (Fig. 1). The average L\*-value of saturated steam treated birch was 52.7. The L\*-value of birch treated in superheated process was 2.8 L\*-units lighter even though process temperature was 25°C higher. No colour difference was observed when outer and inner surfaces from each sample were measured. Difference in a\*-value between the treatments was 1.6 units, indicating slightly more redness after process in saturated steam. Difference less than 0.2 units in b\*-value was observed.

	L*	std	a*	std	b*	std
Reference	80.92	0.97	4.41	0.39	17.01	1,1
Superheated 185°C	55.48	2.15	8.19	0.32	19.89	0.67
Saturated 160°C	52.72	2.20	9.81	0.82	19.73	1.28

**Table 1**. The average colour L\*a\*b\*-values and standard deviation (std).



Fig. 1. The colour L\*-values and standard deviation (std).

#### Equilibrium moisture content and dimensional stability

EMC of untreated birch in RH 85% was 15.2%. The difference to birch treated in saturated steam was 5.0% and even bigger compared to birch treated in superheated steam 6.8%. Dimensional stability was determined by measuring dimensions of samples in three different relative humidity conditions. This test showed that birch treated in superheated and saturated steam performed similarly in all RH (Fig. 2).



**Fig. 2**. Equilibrium moisture content and volume change at T=20°C and three different relative humidity conditions.

#### Acidity

The pH of birch treated in saturated steam (pH 2.90) was lower than the pH of birch treated in superheated steam (pH 3.54). As presented in Table 3. acid content of birch treated in saturated steam was more than three times higher than in superheated steam treated birch.

		Consumed alkali,	(Acetic) acid
Sample	pН	NaOH [ml]	content* [%]
	2.9	43.2	
Saturated 160°C	0		2.90
	3.5	15.7	
Superheated 185°C	4		0.97
-	4.0	-	
Reference	3		0.39

**Table 3.** Measured pH, consumed alkali during titration and acid contents (mainly acetic acid) of thermally modified birch.

\*Acetic acid content was determined on the rough assumption that most of the titrated acid equivalents arise from acetic acid

#### DISCUSSION

There is only small L\*a\*b\* colour value difference between birch treated in saturated steam at 160°C and superheated steam at 185°C. The difference is less than three units in lightness (L\*) and 1.5 units in redness (a\*). EMC of thermally modified birch with both the treatment methods was significantly lower than EMC of untreated birch. The difference in EMC between untreated and thermally modified birch increases with higher relative humidity (RH). Birch modified in saturated steam at 160°C is clearly more acidic compared to birch modified in superheated steam at 185°C. Some preliminary bending strength and Brinell hardness tests were carried out as well. Reduction of bending strength is bigger than expected in saturated steam process. This might have the consequence that the heat treatment temperature should be decreased and the operating moisture content of wood optimized. Any significant difference in hardness was not detected.

#### CONCLUSIONS

Both the thermal treatments carried out in either superheated or saturated steam in this study result in several positive properties. First of all improved dimensional stability and weather resistance are the desired characteristics. But there are also properties which are requiring compromises and optimization regarding the process and parameters used during the process. Thermal modification in saturated steam produces more acidic birch compared to thermal modification in superheated steam. This might have consequences, requiring more research e.g. concerning surface treatment and fixation.

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# A TWO-STEP WOOD PROTECTION PROCESS USING ALTERNATIVE WOOD PROTECTION AGENTS IN COMBINATION WITH AN OIL TREATMENT

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#### ABSTRACT

In this study, natural polymers were tested as possible alternatives for conventional wood preservative in a two-step process. Scots pine sapwood blocks were impregnated with chitosan, tannin, propiconazole and Wolmanit and oil-treated afterwards with a modified linseed oil. Two different fixation parameters were performed. The treated samples were leached according to EN84. The outcome of trials shows that a two-step process reduces the leaching of the main active components. After leaching, the samples were exposed to fungal attack by *Coniophora puteana* and *Trametes versicolor* according to EN113. Mycological tests showed that most of oil treated samples were effective against wood decay.

Key words: chitosan, propiconazole, fixation, fungi test

#### INTRODUCTION

The combination of an impregnation process using a wood protection agent with a following treatment with modified natural oil was developed 31 years ago (Häger 1980), usually known as the Royal process. This process combines the fungicidal properties of a wood protection agent with the hydrophobical properties of oil in a twostep process. The wood preservative used in the Royal process is a water-based chromium-arsenic free preservative (commercial name Wolmanit CX-8). Modern copper-based preservatives have a lower fixation rate compared to CCA, and can easily be leached out during outdoor exposure conditions (Habicht et al. 2003). A combined impregnation process (CIP) has a great environmental advantage; it significantly reduces the leaching of copper in use (Treu et al. 2003).

The use of metal-based wood preservatives is facing increasing environmental and disposal concerns. Organic biocides and some natural polymers with ability to minimize fungal attack are considered as future wood preservatives (Evans 2003).

Chitosan, a natural polymer, is a derivate of chitin, which is manufactured primarily from waste products of the food industry - crustaceans such as shrimps, crabs and crayfish (Brine et al. 1991). In recent years chitosan has received attention as a potential eco-friendly wood preservative (Alfredsen et al. 2004).

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Propiconazole is a derivate of triazole, organic biocide, and is used in wood protection chemicals, because of good antifungal effectiveness and triazoles are biodegradable in soil (Buschaus et al. 1995).

Tannins are natural phenolic polymers, commercially produced from wood and barks. Several observations have shown fungicidal effect of tannins (Scalbert, 1991). It is known that tannins show poor fixation. They accumulate on the wood surface and leach easily (Sen et al. 2009).

The aim of this study was to investigate new potential alternative wood protection agents for a two-step process and to improve fixation by means of oil a subsequent oil treatment and evaluate the performance against fungal attack in lab trials.

# MATERIAL AND METHODS

#### Wood samples

Scots pine sapwood (*Pinus sylvestris* L.) blocks (50 x 25 x 15 mm) were selected, endsealed and oven-dried at  $103 \pm 2^{\circ}$ C for 24 hours. Absolute dry weight was taken and samples were conditioned at 20°C and 65% relative humidity before impregnation.

#### Wood protection agents

Solution	Concent. [%]	Description	Active agent	Chemical analyses				
Wolmanit CX-8	4.0	Commercial wood preservative, $pH = 9.5$	Cu	ICP				
Scanimp	5.1	Commercial microemulsion, pH= 3.0	Propiconazole	ICP				
Kitoflokk	5.0	Chitosan, natural polymer produced from crabs, pH= 5.3	D- glucosamine	HPLC				
Tannin	5.0	Water soluble polyphenol produced from mimosa bark, pH= 4.7						
Oil	-	Modified linseed oil produced from flax plant seed, drying oil						

**Table 1:** Overview of wood protection agents used in a two-step process

The preparation of the chitosan solution, determination of the degree of deacetylation and the molecular weight were examined by methods described by Larnøy et al. (2006). Kitoflokk powder was dissolved in deionized water. Acetic acid was added until pH range 5 to 5.5 was reached. Mimosa tannin powder was dissolved with deionized water without any additional chemicals.

## Two-step protection system

The two-step wood protection system included two process steps: 1. impregnation procedure where wood samples were impregnated with a wood protection solution for 30 min at 40 mbar vacuum and 1 hour at 9 bar pressure. 2. Oil process - in the second step the wood samples were treated with hot oil (modified-linseed oil) at temperature 80°C in a vacuum (100 mbar) for 3 hours. The samples were pulled out from the oil some seconds before the end of the process and air was released in afterwards in order to avoid high oil uptakes of the wood samples. Wood samples were either exposed to hot oil directly after impregnation or stored 24 hours for fixation. Samples without oil treatment were tested as controls. After the two-step protection process, samples were dried at a temperature of 55°C and using 20 mbar vacuum until stabilization (7 days) to determine the oil uptake.

#### Leaching

The conditioned samples were leached according to the European standard EN84 (1997), by impregnating (40 mbar for 20min) wood samples with deionized water to accelerate ageing of the samples. Collected water samples were analyzed for the amount of main active ingredients (Table 1). Tannin was not analyzed.

#### **Decay test**

Samples were vacuum dried after leaching. The specimens were exposed to fungi according to the EN113 (1996) using brown-rot fungi Coniophora puteana (CP) and white-rot fungi Trametes Versicolor (TV).

#### **RESULTS AND DISCUSSION**

Table 2 shows the retention of preservatives and oil. The average uptake for CX-8 was  $25.2 \pm 3.2$  kg/m<sup>3</sup>, which is as twice high as the uptake achieved by a "Lowry process". Samples directly exposed to hot oil after impregnation process had higher oil retention compared to samples with 24 hour fixation. According to previous studies of CIP, the oil uptake increases with increasing fixation time. Higher oil uptake of wood samples without fixation could be explained by cracks that developed due to faster drying on end-sealed surfaces (Treu et al. 2008).

able 2:	Mean retention of	t solutions a	ind mod. linseed oil.
Solution	Retention [kg/m <sup>3</sup> ]	Treatment	Oil retention [kg/m <sup>3</sup> ]
CX-8	25.2(2.2)	directly	151 (43)
	23.2 (3.2)	24 h fixation	63 (25)
ScanImp	24.8(1.2)	directly	207 (54)
	34.0 (1.2)	24 h fixation	75 (17)
Vitoflokk	22.2(4.4)	directly	110 (40)
KITOLIOKK	55.2 (4.4)	24 h fixation	75 (29)
Tannin	221(50)	directly	102 (37)
	32.1 (5.0)	24 h fixation	62 (13)

1...

#### Leaching

When comparing leached main active ingredient, the lowest amounts were released from samples treated in oil directly without 24 hours fixation of the protective solution (Fig. 1-3), except samples treated with Kitoflokk. The amount of leached glucosamine was unexpectedly low, total amount for samples without oil treatment was 2.2 mg/L. Values for oil treated chitosan were < 0.001 mg/L. According to the leaching results, the degree of fixation is very high for all treatments.

#### **Decay test**

The mass loss during fungal exposure is displayed in Fig. 4-7. Samples treated with CX8, Scanimp and their oil combinations showed less than 3% mass loss for both fungi species. According to other studies, chitosan and tannin have fixation problems (Sen et al. 2009). This could not been proved by this study. However, chitosan treated samples without oil show poor protective properties when exposed to brown rot. Furthermore,

tannin- and chitosan- treated samples without oil show poor protective properties against white rot. Chitosan and tannin samples treated in oil directly after impregnation with a wood protection agent and after 24 hour fixation showed very high antifungal effect against CP.

However, chitosan and tannin treated samples without oil treatment showed low antifungal effect against TV.









**Fig. 1-3**. Amount of main active component in leaching water during 14 leaching days. Values for oil treated chitosan < 0.0001 mg/L are not shown in the graph: Fig. 1 - copper (Total CX8= 89.4 mg/L, CX-8+oil= 11.9 mg/L, CX8(24h)+oil= 41.9 mg/L); Fig. 2 – propiconazole (Total Scan.= 21.9 mg/L, Scan+oil= 3.8 mg/L, Scan(24h)+oil= 5.2 mg/L); Fig. 3 – glucosamine (Total Kitoflokk= 2.2 mg/L).







**Fig. 4-7.** Mass loss of different treated and untreated wood samples after 16 weeks of exposure to brown rot (*Coniophora puteana*) and white rot (*Trametes versicolor*). U-untreated sample, US-virulence with end grain sealing, USW- virulence without sealing.

#### CONCLUSIONS

The two-step process significantly reduces leaching of the wood protection agents. The two tested commercial wood preservatives alone or in combination with an oil treatment showed high resistance against fungal attack.

The natural product chitosan showed low resistance against fungal attack. However, in combination with an oil treatment a high resistance against brown rot attack could be shown. In contrast, white rot attack could not be prevented with chitosan in combination with oil.

Wood samples treated with the natural product tannin and in combination with an oil treatment showed good antifungal properties when exposed to brown rot. However, white rot attack could not be prevented. Tannins and chitosan used as a wood protection agent in a two-step process, might be therefore not be suitable as an alternative to CX-8 in CIP.

Scanimp provides high antifungal effectiveness, low leaching, and as an organic biocide could be an alternative product for copper-based products used in CIP.

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# LEACHING OF COMMONLY USED IMPREGNATION AGENTS AFFECTED BY WOOD PROPERTIES

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# ABSTRACT

The objective of the study was to assess the influence of wood properties on copper leaching from wood treated with preservatives.

Scots pine (*Pinus sylvestris* L.) trees were harvested from two different stands in Norway and one in Denmark. Sapwood was cut to samples (20 x 20 x 50mm) in as many layers as the radial size allowed. Within this material, it is possible to trace the individual sample to its original position in the stem. Approximately half of the samples were treated with Wolmanit CX-8 and half with Tanalith. All samples were conditioned, impregnated with preservatives and leached according to EN84. Copper and boron content in water samples was determined by an ICP (Inductively Coupled Plasma) technique. The variation in leachability within trees, between trees and between different stands was studied.

Statistical analyses showed that trees from the south are more prone to leaching and that samples from the lowest part of the tree fixate less preservative than those from the upper parts. In addition, drying method of the sample had an influence and differences were also noted between products used in the study.

Key words: Copper-based wood preservatives, EN84, leaching.

## INTRODUCTION

Wood is a renewable resource with many positive qualities but the wood industry faces challenges due to the changing environmental policies about the use of wood preservatives. Preservatives containing arsenic and chromium have been banned from the market. There have been studies indicating that copper in other systems is not so strongly bound (Habicht et al. 2003, Temiz et al. 2006) and the preservatives used in this study are common copper-based impregnation solutions.

Copper is naturally occurring element, mostly present in surface waters and is an essential micronutrient for plants and animals at low concentrations (Kapustka et al. 2004). At elevated concentrations, it may become toxic to aquatic life forms. Toxicity concentrations are species and water body dependent (Aquatic life ambient... 2007).

The objective was to assess the dependence of leachability on different wood properties of Scots pine. Previous studies have been made on permeability of wood that used the same raw material (Larnøy et al. 2008, Lande et al. 2010, Zimmer et al. 2009). It was

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found that treatability was affected by latitude, the origin of the sample with respect to vertical and horizontal stem position, tree height and method of drying.

# MATERIAL AND METHODS

#### Wood samples

Three Scots pine stands were chosen for this study, two from Norway and one from Denmark. Nine trees were harvested from each site, 27 trees in total. After removing the top of the tree, the stem was divided into 5 sections of 60 cm. Sections were taken from 0, 25, 50, 75 and 100% of remained tree height. From each of the section a block of 75 cm width with north-south orientation was sawn. Blocks were split exactly through the pith. The two halves of each block were either "air dried" or "kiln dried". Drying method for the north and south pole was randomly selected.

Only sapwood was chosen for this study, heartwood was removed. Samples with dimensions of approximately  $20 \times 20 \times 50$  mm were cut out in as many layers as the radial size allowed. In total, 931 samples were tested.

After cutting, samples were conditioned at 65% RH and 20°C until equilibrium moisture content was reached. To prevent the liquid flow in the longitudinal direction, cross sections of the samples were sealed with a two component sealer (Pyrotect-2K-Aussen-Schutzlack 1720-7100-302, Dreisol coatings GmbH, Germany).

This set of samples was impregnated with Wolmanit CX-8 and Tanalith, which are two widely used wood protection solutions.

478 samples were treated with Wolmanit CX-8, which is a chromium free, copper- and boron-based wood preservative consisting of 2.8% Bis-(N-cyclohexyldiazeniumdioxy)-copper (Cu- HDO), 13.04% Copper hydroxide carbonate and 4% boric acid.

459 samples were impregnated with Tanalith, which is a water-borne wood preservative based on copper and co-biocides consisting of 22.5% Copper Carbonate, <45% 2-AminoethanolCarbonate, 0.49% Tebuconazole, 4.9% Boric Acid and <5% Di-2-ethylhexylphthalate.

#### Impregnation

Impregnation was performed at 6 bar for 10 min. Wood samples were weighed immediately after impregnation to calculate respective uptake values.

#### Leaching

Test samples were conditioned in the same way as it had been done before impregnation (65% RH and 20°C till equilibrium moisture content).

Leaching was done according to EN84. Samples were covered with deionized water in an amount of approximately five times the volume of the sample and placed in the impregnation vessel. Samples were held in 0.04 bar of vacuum for 20 min. After vacuum, the samples stayed in the water for 2 h before the water was changed for the first time. Specimens were submersed in deionized water for 14 days. From every sample`s vessel, 5 ml of leaching water was collected, combined and submitted for chemical analyses. Water changes and collecting of water samples were done ten times (including the water change after impregnation in 2 h).

**Table 1:** Schedule for taking leaching samples.

Day	1	2	3	4	5	6	7	8	9	10	11	12	13	14	
Amount (ml)	5 5	5	-	-	5	5	5	5	5	5	-	-	-	5	50

#### Chemical and data analysis

Elements of interest in the leaching water were copper and boron. For tracing the amount of leachates from the samples, the leaching water was analyzed via ICP (Inductively Coupled Plasma) technique. For ICP the water samples were neutralized with hydrochloric acid (HCl).

To discover the sources of variance, results of leach-outs were correlated with different physical and anatomical wood properties. To determine the treatability of the wood samples and to find relations to leaching, ratio of filling (RoF) was calculated (Eq.1, 2), which relates the filled volume to the void volume that can be filled. It shows how much of the possible void volume is actually filled with liquid (Larnøy et al. 2008).

$$V_{\text{void}} = V_{12\%} - V_{\text{cellwall}} - V_{\text{water}} + V_{\text{swell}}$$

$$RoF = V_{\text{uptake}} / V_{\text{void}} \times 100 \quad [\%]$$
(1)
(2)

 $V_{void}$  is the void volume in wood  $V_{12\%}$  is the volume of the sample at 12 % MC  $V_{cellwall}$  is the volume of the cell wall material  $V_{water}$  is the volume of water at 12 % MC  $V_{swell}$  is the potential increase in volume due to swelling  $V_{uptake}$  is the volume of the treating liquid

Variables used are summarised in Table 2.

Variables	Abbr.	Туре	Comment
Location	Loc	nom	- Denmark - Norway, S-E - Norway, mid-E
Latitude	Lat	cont	56°- 61° North
Tree ID	Т	nom	27 trees
Tree height	TH	cont	12-27 m
Tree age	ТА	cont	18-300 years
Exposure side	ES	nom	North or South side of log
Diameter at breast height	DBH	cont	15-46 cm
Sample Height	SH	cont	0.3-20 m, 1-5 layers from the stem
Radial position	RP	cont	Registered as year after heartwood
Density	D	cont	393 -696 kg m <sup>-3</sup>
Annual ring width	AR	cont	0.4-3.1 mm
Method of drying	MDr	nom	- Kiln dried at 60 °C (a) - Air dried at 20 °C (n)
Sample layer	SL	nom	1-4 layers of sapwood
Impregnation liquid	IL	nom	- Wolmanit CX-8 (W) - Tanalith (T)
Cu leachout	CuL	cont	Response variable

**Table 2.** Overview of variables used in the study.

Statistical analyses were executed with JMP Pro 9 by SAS Institute Inc.

When promising variables were discovered, a linear mix model was generated. Linear mix model subsumes different variables simultaneously. The parameter estimates show how much the variable contributes to the response and the test statistics expresses if the effect is significant.

For the present model (Table 4), variables that showed correlations or significant difference when describing leaching were used as fixed effects. Individual tree was taken as a random effect meaning that the effect is selected by chance from a larger sample pool.

The mixed model can be written

 $Y = \mu + T_i + \texttt{l}_D + \texttt{f}_1 + \texttt{a}_a + \texttt{b}_T + \texttt{g}(RoF) + \texttt{e}_{iD1aT}$ 

## **RESULTS AND DISCUSSION**

Variations influenced by raw material in sapwood penetration have been found in former studies (Larnøy et al. 2008, Lande et al. 2010, Zimmer et al. 2009), which could mean that leaching of preservatives is also affected by wood properties.

Some samples (both Wolmanit CX-8 and Tanalith treated pieces) were regarded as outliers according to Mahalanobis and Jackknife Distances and, therefore, excluded from the analysis.

Treatment	*	Dimension	Ν	Mean	Std dev	min	max
Wolmanit CX-8	uptake	kg m <sup>-3</sup>	471	210	38	114	312
Tanalith	uptake	kg m <sup>-3</sup>	448	209	32	109	290
Wolmanit CX-8	RoF	%	471	26	5	13	39
Tanalith	RoF	%	448	26	4	13	39
Wolmanit CX-8	Cu leaching	$mg L^{-1}$	471	0.58	0.16	0.30	1.14
Tanalith	Cu leaching	$mg L^{-1}$	448	0.74	0.24	0.33	1.43
Wolmanit CX-8	B leaching	mg L <sup>-1</sup>	471	0.57	0.14	0.28	0.93
Tanalith	B leaching	$mg L^{-1}$	448	0.43	0.11	0.16	0.78

**Table 3:** Summary of outcomes

One purpose of this study was to observe how the permeability of the material is correlated with the leaching of the preservative. To get a better overview of the impregnation volume, impregnation schedule was intentionally too short for full impregnation and samples were not completely filled. Wolmanit CX-8 and Tanalith are equally distributed within the samples: both treated samples are filled approx 13-39 % (Table 3). When correlating RoF to leached out Cu amount, the results show that there are not very strong relations ( $R^2 \sim 0.34$ ) which suggest that the wood properties have an influence. There is a slight trend indicating that larger amount of Cu and B leach out at higher uptakes of preservatives. This influence was more pronounced in the case of B ( $R^2 \sim 0.69$ ).

The conversion from mg L<sup>-1</sup> into %-s, it shows that approx 18 % of Cu leaches out from Wolmanit CX-8 samples and 23 % from Tanalith samples. Almost all of the boron leached out from the samples, independent whether they had been treated with Wolmanit CX-8 or Tanalith. It is also mentioned in the literature (Waldron et al. 2005, Ibach 1999) that boron fixates poorly in the wood and therefore it is difficult to estimate how much of the wood properties actually influence the leaching of B.

The results of the correlation between the abovementioned variables (Table 2) and the total amount of leach-out, indicates the most important parameters for leachability. Latitude, samples` vertical tree position and drying method suggest influencing leachability. Samples coming from the southern forest sites, the lowest part of the tree and that were kiln dried tended to leach out the most preservative. Lande, Larnøy and Zimmer (2008-2010) who studied treatability of Scots pine using the same stands as the present study, found that the lowest tree sectors from the southern stand is the easiest to treat.

A linear mixed model employing method of drying, impregnation liquid, latitude and tree height as fixed effects and tree as a random effect explains approximately 44% of the variation in Cu leaching.

$\mathbf{R}^2$			0.44	
Random effects	Parameter	Var.Component		Pct of total
Tree	T (i=1-10)	1.42		5.54%
Residual	е	24.25		94.46%
Fixed effects		Estimates	F-ratio	Prob> t
Method of drying	a <sub>a</sub>	-1.74	38.15	<.0001
	an	1.74		
Impregnation liquid	bw	-2.86	114.65	<.0001
	b <sub>T</sub>	2.86		
RoF	g	-0.43	30.37	<.0001
Latitude	lD	1.45		
	ls	0.27	6.11	0.0032
	$l_{\rm H}$	-1.72		
Height	$f_1$	3.18		
	f <sub>2</sub>	-1.12		
	f <sub>3</sub>	-0.85	11.48	<.0001
	£ 4	0.47		
	f <sub>5</sub>	-1.68		
Intercept	μ	30.68		<.0001

**Table 4.** Model parameters to predict Cu leachout (%)

In this model, the factor "impregnation liquid" has the biggest impact on leaching. This trend was also seen for Wolmanit CX-8 and furfuryl alcohol (Larnøy et al. 2008). This might be explained by the fact that furfuryl alcohol and Wolmanit CX-8 differ more in molecular size than the solutions used here.

The drying method is also of significance. Previous studies have shown that higher drying temperature gives higher penetration values which might be due to opening of the radial and longitudinal resin canals (Booker 1990) or evaporation of volatile extractives (Lande et al. 2010). This could in turn mean that higher temperatures during kiln drying alter the chemistry of the cell wall and reduce the fixation of preservatives as compared to air dried timber.

## CONCLUSIONS

The study shows the influence of the harvesting site and wood variables on the leaching behaviour of copper-based preservatives.

- 1. Samples taken from southern stands have higher leaching values than those from northern ones.
- 2. A significant correlation between vertical stem position of the samples and the emission of Cu and B from preservatives was exhibited. Preservatives leached more out from lower than from higher parts of the stem.
- 3. Samples that were air dried leached out less preservative than the ones that were kiln dried.

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# SURVEY OF RESEARCH PROJECT: ENHANCED SERVICE LIFE OF COATED WOODEN FACADES

# Hovde, P.J.<sup>1</sup>

#### ABSTRACT

During the period 2007-2011 a research project has been carried out in cooperation between Norwegian universities, research institutes and industry. The main objective is to develop new methods for early prediction of durability and longer aesthetic service life of outdoor coated wooden claddings. Various test specimens from Norway spruce (*Picea abies* (L.) Karst) with surface treatment have been exposed to natural (outdoor) and artificial (laboratory) weathering. Two laboratory methods, qPCR and ATR-FTIR spectroscopy, have been studied as tools for qualitative and quantitative identification and evaluation of early colonization by discolouring fungi on painted surfaces. The methods are also compared with other existing methods for evaluation of discolouring fungi. The wood materials used in the project are carefully selected and characterized, in order to detect any influence on the growth of discolouring fungi by the wood structure, growth conditions and wood material position in the timber log. Finally, a survey is made of service life prediction methods and how such methods have been applied on wooden building products.

Key words: facades, wood, fungal growth, durability, service life

#### INTRODUCTION

#### **Project description**

The project started in 2007 and will run until the end of 2011. The main objective is defined as "Develop new methods for early prediction of durability and longer aesthetic service life of coated outdoor wooden claddings related to consumer needs and new building and environmental regulations." The research work of the project is organised in 7 Work Packages (WP), and the titles should explain the content of the WPs and the overall work to be done in the project.

#### **Cooperation partners and funding**

The project is carried out in a joint cooperation between the following partners:

> Norwegian University of Science and Technology (NTNU), project management

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- Norwegian Forest and Landscape Institute (Forest and Landscape)
- > Norwegian Institute of Wood Technology (NTI)
- Norwegian University of Life Sciences (UMB)
- > SINTEF Building and Infrastructure (SINTEF)

Two industry partners are involved in the project. Jotun AS and Kebony ASA have supplied surface treatment and modified wood materials (furfuryl alcohol treated), respectively. The project work is mainly funded by the Research Council of Norway. In addition, funding is received from The Norwegian Sawmilling Industry, Viken Skog BA, and the Wood Research Fund at the Norwegian Institute of Wood Technology. The research institutions involved in the project are also contributing to the project work by various ways of funding and support.

# PROJECT ACTIVITIES AND OUTCOME

#### General

This paper gives a very brief survey of the work packages of the project. The research work has been carried out by the partners as mentioned above, and the results have been published internationally in conferences, seminars or scientific journals. Only published papers are referenced in this paper. However, activities in most of the work packages are still going on, and some more publications will appear at the end of the project. The project is also presented in an Internet website, www.woodfacade.com.

## WP1: Method development for evaluation of fungi – qPCR

**<u>Objective</u>**: To establish the qPCR method as an objective tool for quantitative and qualitative identification and evaluation of early colonization by discolouring fungi on painted surfaces, in the paint matrix and in wood.

The work of this WP has been carried out at Forest and Landscape, partly in cooperation with UMB. The work has been focused on development of quantitative polymerase chain reaction (qPCR) as a method for early detection of fungi on painted wooden surfaces. qPCR is based on analysis of DNA sequences from species. Adaption of the method for analysis of fungi has been a quite challenging task. The development of qPCR and taxon-specific primers has provided new possibilities for specific detection and quantification of fungi in their natural substrates. The work has produced adjusted DNA isolation protocols and optimized real-time PCR assays for species specific detection of fungi frequently found on painted surfaces (*Aureobasidium pullulans, Alternaria alternata, Cladosporium cladosporides, Ulocladium atrum*).

Results from WP1 are presented in Larnøy et al. (2010) and Larnøy et al. (2011).

## WP2: Method development for evaluation of fungi - FTIR

**<u>Objective</u>**: To establish FTIR spectroscopy as an objective tool for quantitative and qualitative identification of early colonization by discolouring fungi on painted surfaces.

The work of this WP has been carried out at NTNU and SINTEF. Fourier Transform Infrared Spectroscopy (FTIR) is a widely used spectroscopy method for qualitative and quantitative analysis of the chemical composition of substances. Atoms or atomic groups in molecules are identified due to the vibrations of these atoms or atomic groups when irradiated by infrared light at different wavelengths, normally in the interval 4000 cm<sup>-1</sup> to 400 cm<sup>-1</sup>. Performing a FTIR analysis yields a "fingerprint" spectrum specific to the actual material specimen, and changes due to ageing processes may be studied. That is, decomposition and formation of chemical bonds and products can be investigated in a FTIR analysis, and both qualitative and quantitative FTIR measurements may be performed. In this project, work has been done on FTIR analysis of different wood species (both untreated and treated) which have been exposed to natural as well as artificial climate conditions. Analysis is also performed on the same fungi species as mentioned for WP1. Important issues to be studied are how climate induced degradation of wood substance can be detected, how different fungi species can be identified, and if and how the FTIR spectrum of a fungus is influenced by the substrate acting as nutrition for the actual fungus.

Results from WP2 are presented in Jelle et al. (2008a), Jelle et al. (2008b), Jelle et al. (2008c), Gupta et al. (2010) and Gupta et al. (2011).

## **WP3:** Comparison of evaluation methods

**<u>Objective</u>**: To perform comparative studies of different methods for quantitative evaluation of discolouring fungi.

The work of this WP has been carried out at Forest and Landscape and UMB, in cooperation with the other partners. Today, a number of evaluation methods to describe the degradation or discolouration of treated or untreated wood have been standardized. These methods are qualitative, and fungal growth on the surfaces is evaluated according to different visual inspection methods. In this project, wood specimens with the actual surface treatment applied in the project have been exposed to different types of fungi under laboratory conditions (see WP1). Similar specimens exposed in natural climate conditions are also studied. For specific evaluation of an existing method, the procedures described in NS-EN 927-3 (2000) are applied. That includes visual inspection, scanning of the specimens and taking photos. The fungal growth on the painted wood specimen surfaces is evaluated according to a scale from 0 to 5. One important task is to compare the evaluation results with the test results from WP1 (use of qPCR) and WP2 (use of FTIR).

Results from WP3 are presented in Gobakken (2010).

## WP4: Natural and artificial weathering of wooden claddings

**<u>Objective</u>**: To establish methods and procedures at different laboratory scales for artificial weathering of wood materials, in order to simulate natural climate exposure and material degradation, and to establish acceleration factors.

The work of this WP has been carried out at all the project partners. Degradation and durability of building and construction materials due to climate exposure can be studied according to two different procedures: natural exposure at different locations with
different climates, or under artificial laboratory conditions including few or more climate factors (temperature, humidity, UV radiation, biological factors). The work of this WP includes both natural exposure of surface treated wood materials and artificial laboratory exposures. Outdoor testing has been performed in Ås, in Sørkedalen outside Oslo and in Trondheim, and this covers some different climates. In the laboratory, QUV testing has been carried out at NTI, whereas NTNU/SINTEF have been testing similar specimens in a climate carousel (NT Build 495) and an Atlas solar simulator. The exposed materials have been used to prepare specimens for specific studies performed in most of the other WPs.

### WP5: Characterization of wood materials – implications for fungal growth

**<u>Objective</u>**: To characterize all wood used in the project in order to provide a reliable basis for the statistical analyses performed in the different WPs, and to discover effects of wood properties on the performance of coatings.

The work of this WP has been carried out at UMB and NTI. It is well known that the performance of wood in outdoor applications depends on the wood quality as well as surface treatment or impregnation. The influence on wood-coating interactions on the growth of discolouring fungi is still not well enough explored. In many studies of coated wood, the description of the wood is sparse and the variation between the wood specimens is seemingly of little interest. However, since wood is a natural material which can be characterized as both non-homogenous (heartwood and sapwood, knots) and anisotropic (fibre orientation, knots), it is important to study how different growth conditions will influence on degradation and durability properties. The wood species studied was Norway spruce (Picea abies (L.) Karst), and in order to have different levels of annual ring width, materials were sampled from two fertile sites and two poor sites in South-eastern Norway. Butt logs and second logs were processed to claddings, and the claddings to be analyzed were chosen from both inner centreboards and outer centreboards. To see the effects of the timber log structure and the growth conditions, both radially sawn claddings as well as tangentially sawn claddings were made. A careful characterization of the materials and some key properties has been carried out.

Results from WP5 are presented in Sivertsen (2010) and Sivertsen and Vestøl (2010).

### WP6: Characterization of coatings and wood performance

**<u>Objective</u>**: To find the best combination of wood material, surface condition and treatment to achieve longer maintenance intervals of painted claddings.

The work of this WP has been carried out at all the project partners. The work of this WP comprises inspections, measurements and analysis of the degradation of the actual wood materials and treatments due to the natural or artificial climate exposures described in WP4. The outdoor exposed materials were inspected twice a year, namely in May/June and in September/October, respectively. The main part of the work comprises visual inspections of the outdoor and laboratory exposed materials, and measurement of moisture uptake in fresh and artificial laboratory (QUV) exposed materials. An important issue is to study relations between wood characteristics, moisture uptake and paint and wood degradation due to the growth conditions and timber log structures as mentioned for WP5.

Results from WP6 are presented in Gobakken et al. (2008) and Gobakken (2010). WP7: Service life prediction of wooden claddings

<u>**Objective</u>**: To establish a procedure for aesthetic service life prediction of exterior wooden claddings due to climate exposure.</u>

The work of this WP has been carried out at NTNU and Forest and Landscape, in cooperation with the other partners. During the project work, it turned out that the outcome of the project does not supply sufficient type and amount of data to perform a service life prediction of the materials used. Instead, a survey is made of the state-of-the-art of service life prediction methods and the application of such methods to wood materials and components in buildings.

Results from WP7 are presented in Listerud et al. (2011).

# CONCLUSIONS

- These conclusions only give a very brief survey of the main activities of the project. Detailed results are presented in the publications given in the reference list.
- The analytical method quantitative polymerase chain reaction (qPCR) has been further developed for determination of discolouring fungi on coated wooden claddings. Practical application of the method compared to more traditional evaluation methods is studied.
- Application of Fourier transform infrared (FTIR) spectroscopy to study degradation and durability of treated and untreated wooden claddings as well as growth of discolouring fungi on such materials has been studied carefully. Much experience is gained regarding the method.
- ➤ A standardized evaluation method for durability (?) of wood materials has been studied and compared to the qPCR and FTIR methods developed in the project.
- ➤ Wood materials for use in the project have been carefully selected regarding growth site and conditions and position in the timber log. The materials have been characterized regarding key material properties in order to see how these properties influence on the degradation and durability of surface coating and on discolouring fungal growth on the coating.
- Treated surface claddings have been tested at outdoor climate exposure at different locations in Norway (Ås, Oslo, Trondheim). The same materials have been tested at artificial laboratory conditions to study the degradation and durability and the effect of different climate parameters. Careful visual inspections of the materials have been performed twice a year to characterize the type and extent of damage and degradation. Inspections and measurements are also performed on the laboratory exposed materials.
- The project did not supply sufficient data to perform service life prediction of the applied materials. A survey of service life prediction methods and the application on wooden building materials has been made.

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# THE EFFECT OF WOOD PROPERTIES ON THE NATURAL WEATHERING PERFORMANCE OF COATED CLADDINGS MADE OF NORWAY SPRUCE

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### ABSTRACT

The influence of wood properties on cracking, blistering, and flaking of a 3-layer acrylic coating system was investigated in a natural weathering test from autumn 2007 to spring 2011. The performance of the coating was mainly reduced in the lowest third of the test samples. The origin of the samples in terms of vertical stem position did not have an influence on the performance of the coating. The coating system exhibited less cracking and blistering on samples with standing year rings, i.e., quarter-sawn boards with growth rings running parallel with the narrow sides of the board. Blistering was most severe on samples where the pith side was coated.

Key words: cladding, coating, natural weathering, outdoor exposure, service-life

### INTRODUCTION

Coated wooden claddings have a long tradition and still are the most popular exterior wall material in Scandinavia. The competition from other materials that require less maintenance has been increasing over the years, and poses the challenge for the wood and the related coating industry to develop strategies to enhance service-life of claddings made of wood. The ultimate objective to develop products with no need for maintenance is probably unrealistic to obtain from today's point of view but a minimum of 10-15 years intervals is in the realms of possibility.

The performance of a coating is related to its chemical composition, climate conditions, the wood properties, and construction details of the cladding. In recent years, industrial coating has become more common and water-borne coatings prevail on the marked. The primer is usually an alkyd of low molecular weight to provide sufficient penetration in the wood surface while the top coat is an acrylic dispersion or a combination of an acrylic dispersion and alkyd emulsion. These top coats stay quite elastic and, thus, show a less tendency to crack than pure alkyd systems that had been used in many years. Besides, cracking and other coating failures also depend on wood properties, as for instance density and ring width (Williams 2010), ring orientation (Feist and Hon 1984; Flæte et al. 2000), heartwood content (Jämsä 2006), knots (Williams 2010), moisture content (Graystone 2004), and surface roughness and chemistry (Hora 2001).

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In the present study, possible effects of some wood properties on cracking, blistering, and flaking of a 3-layer acrylic coating system were investigated during natural weathering between autumn 2007 and spring 2011. The study is a part of the project "Enhanced service life of coated wooden facades" (The Research Council of Norway, project no. 178325), which aims to develop new methods for early prediction of durability and for longer aesthetic service life of coated wooden claddings made of Norway spruce (*Picea abies* (L.) Karst.).

### MATERIAL AND METHODS

The wood material was sampled in 2 regions, Toten and Larvik, of different growth conditions in southern Norway. Spruce trees from Toten were slowly-grown from 2 stands with low site indices as dominant height at 40 years age (H<sub>40</sub>) of G8 (N60°40'49,8''; E10°31'47,8'') and G11 (N60°42'08,9''; E10°32'00,2''). Those from Larvik were fast-growing trees from 2 stands with site indices H<sub>40</sub> of G23 (N59°04'07,8''; E9°57'16,5'') and G26 (N59°04'06,9''; E9°57'06,6'').

Trees from Larvik were cut into approx. 5 m long pieces while those from Toten were cut into an approx. 4 m long log from the lower part of the stem and an approx. 5 m long log from the upper part of the stem. Two flat sawn planks (1 and 2) were cut out from one half of the round blocks. From the other half, a plank (3) with "standing" year rings (s) were sawn (Fig. 1). Afterwards, the planks were kiln-dried and then split and sawn to 2 boards each. The test comprised in total 269 boards (19 x 98 x 80 cm<sup>3</sup>; t x w x l), which were grouped into 4 categories according to the side where the coating was applied on:

m = flat-sawn boards (of plank 1) with the pith side coated,

y = flat-sawn boards (of plank 1) with the side coated pointing towards the bark,

l = flat-sawn boards with "lying" year rings (of plank 2) with the side coated pointing towards the pith,

and s = quarter-sawn boards (of plank 3) with "standing" year rings.



Fig. 1. Origin of the samples in the logs.

The boards were industrially coated. The coating system consisted of an alkyd-based primer, a red acrylic-based mid- and topcoat of the company JOTUN A/S.

The film thickness was measured by microscopy on 5 boards. Three bars were cut out from each board perpendicular to the grain. Three samples were analyzed from each bar, resulting in a total of 45 samples.

The performance of the coating against natural weathering was tested on TRETEKNISK's test field in Sørkedalen, approx. 10 km west of Oslo. The samples were exposed in July 2007 and evaluations were carried out in spring and autumn from 2008 to 2010. The last evaluation was taken in spring 2011. The test procedure followed EN 927-3:2006 with some deviations concerning sample preparation and evaluation: the ends of the samples were not sealed. Each panel was divided into 3 sections of same size (A-C) that were separately evaluated. The rating scheme for the designation of cracking, blistering, and flaking was scaled from 0 (not visible) to 5 (many and very large cracks) in steps of 0.5.

### RESULTS

The 3-layered coating system was in average 182  $\mu$ m (STDEV of 25  $\mu$ m) thick. The mean of the board with the thinnest film was 159  $\mu$ m, the mean of that with the thickest film 211  $\mu$ m.

The first very small cracks, which penetrate the whole coating system, of class 0.5 were observed in spring 2008 (Fig. 2). Cracking became more severe in the following years with maximum ratings of class 3 found in section C, i.e., the lower ends of the samples. Cracking was clearly more pronounced in section C than in the sections A and B during the entire test period.

The vertical stem position, i.e., whether the sample comes from the lower or the upper part of the tree, did not have an influence on cracking. Samples with "standing" year rings (s) exhibited less cracking than the others (Table 1).



**Fig. 2.** Cracking in the cladding samples (A = upper section, B = middle section, C = lower section, mean = whole sample).

Table	1.	Statistics	on	cracking	in	samples	with	different	sides	coated	(see	Fig.	1).
Means	no	ot connecte	ed w	vith same 1	lett	ers are sig	gnific	antly diffe	erent a	t the 95	% co	nfide	nce
level (	Tul	kev HSD t	est).	Note that	th	e data are	stron	gly right-	skewe	d.			

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Evaluation	Side	Min	Qu. 1	Qu. 2	Mean	Qu. 3	Max	Tukey	
								α=0.05	
	у	0.00	0.05	0.14	0.15	0.17	0.64	а	
A 11	m	0.00	0.00	0.05	0.14	0.17	0.79	а	
All	1	0.00	0.05	0.14	0.13	0.17	0.48	а	
	S	0.00	0.00	0.00	0.05	0.09	0.26	b	
	у	0.00	0.17	0.17	0.22	0.17	1.17	а	
Spring	m	0.00	0.00	0.17	0.23	0.25	1.50	а	
2011	1	0.00	0.17	0.17	0.19	0.17	0.83	ab	
	S	0.00	0.00	0.00	0.09	0.17	0.50	b	



**Fig. 3.** Blistering in the cladding samples (A = upper section, B = middle section, C = lower section, mean = whole sample).

level. Note that the data are strongly right-skewed.									
Evaluation	Side	Min	Qu. 1	Qu. 2	Mean	Qu. 3	Max	Tukey	
								α=0.05	
	у	0.00	0.02	0.05	0.13	0.19	1.02	BC	
A 11	m	0.00	0.05	0.24	0.30	0.43	1.64	А	
All	1	0.00	0.00	0.07	0.17	0.26	1.14	В	
	S	0.00	0.00	0.00	0.03	0.02	0.36	С	
	у	0.00	0.17	0.33	0.39	0.50	1.67	В	
Spring	m	0.00	0.17	0.50	0.57	0.83	3.00	А	
2011	1	0.00	0.00	0.33	0.37	0.50	1.67	В	
	S	0.00	0.00	0.00	0.07	0.00	0.50	С	

**Table 2.** Statistics on blistering on samples with different sides coated (see Fig. 1). Means not connected with same letters are significantly different at the 95% confidence level. Note that the data are strongly right-skewed.

The ratings for blistering are higher than those given for cracking (Fig. 3). Poor adhesion was mainly found in section C, i.e., the lower ends of the samples. Blistering was early observed after the samples had been exposed in summer of 2007. This applies especially to knots. In the first evaluation, approx. 25% of all samples were rated to

class 0.5 or 1 in section C. In the following, the number and size of blisters increased clearly. While blistering was least pronounced on samples with "standing" years rings (s), it was most pronounced on those samples where the pith side was coated (m, Table 2). The vertical stem position did not have an impact on blistering.

Flaking had not become obvious before 2009 but then started to develop particularly on knots (Fig. 4). Since autumn 2010, flaking has been more present on section C than on section A and B. Similar to the results on flaking and blistering, the vertical stem origin of the samples was not found to influence flaking. In contrast to the findings for the other coating failures, the field test did not reveal any effect of the sample side that was coated on the development of flaking.



**Fig. 4.** Flaking in the cladding samples (A = upper section, B = middle section, C = lower section, mean = whole sample).

# DISCUSSION AND CONCLUSIONS

None of the coating failures was ranked higher than class 3 until spring 2011. The relatively early development of blistering, however, was unexpected and disagrees with JOTUN's previous experience with the coating system used. It turned out that the primer cured not properly and the middle coat was applied too soon, which most likely resulted in early blistering especially on the knots.

The process conditions, however, were stable during coating and applied to all samples. It is therefore assumed that the observed differences in coating performance are due to the varying wood properties, which were intended to be studied.

The results show that the tendency of coating systems to crack is lowest on samples with "standing" year rings (s), i.e., quarter-sawn boards with growth rings running parallel with the narrow sides of the board. The low cracking is ascribed to swelling/shrinkage in radial direction, which is approx. half of that in tangential direction in Norway spruce. Besides flaking, blistering was less on quarter-sawn boards

(s) than on flat-sawn boards (l, m, y). Though quarter- or rift-sawing can be considered to be the most ideal for claddings, it results in fewer product yields than flat sawing, and it is questionable whether the marked paid the additional costs for such products.

Blistering was most severe on samples where the pith side (m) was coated. Some of the boards included the pith, which seems to be a worse substrate for coating than wood.

The background for testing samples from different heights of the stem was to take differences of knot size and density into account. The test did however not reveal an influence of the vertical stem position on the coating performance. In case of density, this may be ascribed to the low variation in density in spruce (Hundhausen and Høibø 2010).

The distribution of failures was uneven on the samples: cracking, blistering, and flaking were most pronounced in section C, which indicates that the lower parts of claddings are most susceptible to coating failures. Since the end grain of the boards were not sealed in this test, moisture was easily taken up and given off, which caused tension and subsequently cracking. Once a crack is formed, water easily penetrates into the wood. High moisture in the lower ends of the samples also explains the more pronounced blistering and flaking in section C than in the upper sections because water at the interface between coating and wood surface affects the adhesion.

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# FTIR STUDY ON COMPARISON BETWEEN WOODMOULD FUNGI CULTIVATED ON WOOD AND CULTIVATED ON WOOD COATING

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### ABSTRACT

Service life and aesthetics are important for outdoor wooden claddings and building façade materials. Powder like mould fungi grow on wood surfaces and spread, subject to the availability of nutrients, moisture, oxygen and the temperature conditions. The types of nutrients are believed to cause a change in the fungal cell material. There is a need to identify different fungi species as part of service life evaluations of wooden claddings, even if they are influenced by different substrates. The objective of this study is to characterize wood mould fungi cultivated on two different substrates through attenuated total reflection (ATR) - Fourier transform infrared (FTIR) spectroscopy. The mould fungus *Aureobasidium pullulans* was cultivated in laboratory conditions on uncoated and red coated pine wood (*Pinus sp.*). Preliminary data reveal that ATR-FTIR spectroscopy may have potential to be used to identify and evaluate the changes occurring in the fungal cell material harvested from different nutrient sources. Specifically, the fingerprint region between 1200 cm<sup>-1</sup> – 700 cm<sup>-1</sup> is an interesting source for evaluation of fungal cell wall components.

Key words: Wood Material, Building Pathology, Fungus, ATR, FTIR, Aesthetic.

#### INTRODUCTION

Wood mould fungi are considered as serious threats to building inhabitants by changing indoor air and living conditions causing chronic pulmonary diseases, occupational illnesses and pulmonary problems (Cestari et al., 2002;Ceylan et al., 2006). Building fungi that cause mould belongs to *Chaetomium* spp., *Ophiostoma* spp., *Ceratocystis* spp., *Ascomycetes* spp., *Leptographium* spp., *Cephaloascus* spp., *Phialophora* spp., *Alternaria* spp., *Acremonium* spp., *Aspergillus* spp., *Aureobasidium* spp., *Fusarium* spp., *Oidiodendron* spp., *Cladiosporium* spp., *Penicillium* spp., *Stachybotrus* spp., *Trichoderma* spp., *Coprinus* spp. and *Peziza* spp. Mould fungi are powder like material that accumulate on the surface (Gobakken et al., 2010;Gupta et al., 2010;Hippelein and Rugamer, 2004;Hukka and Viitanen, 1999;Isaksson et al., 2010). Like decay fungi,

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mould fungi also reproduce by spores. However, decay fungi cause strength damage while mould fungi cause aesthetic damage (Alfredsen et al. 2004, Eaton and Hale 1993). Additionally, mould fungi facilitate attack by other microorganism on the surface. Many studies have been performed on wood decay and wood decay fungi (Blankenhorn et al., 1980;Cabib et al., 1988;Chaparro et al., 2009;Flournoy et al., 1991). The goal of this study is to evaluate the mould fungi cultivated separately on wood and on coated wood that are used for building façades by Fourier transform infrared spectroscopy.

### MATERIALS AND METHODS

**Materials**: Mould fungi strain, *Aureobasidium pullulans* (DSM 2404, Deutsche Sammlung von Mikroorganismen und Zellkulturen GmbH, Germany) was supplied by the Norwegian Forest and Landscape Institute, Ås, Norway. Pine wood (*Pinus* sp) pieces of dimensions 70 mm x 70 mm x 25 mm were used as substrates. Wood surfaces were coated with red paint (Jotun OPTIMAL) to make specimens for coated wood surface.

**Inocculation**: Fungi were cultivated initially in on agar medium prepared by 6.25 g Bacto malt, 0.5 l of ionized water and 10 g NMD agar (NMD = Norsk Medisinal Depot AS). Approximately 50 ml of sterilized water having 0.02 % Tween 80 (Merck, Germany) were used for making blend with fungal spores. The aqueous blends containing fungal spores were filtered through cotton pads in a 100 ml Erlenmeyer flask. One drop of the fungal spore suspension was poured on a counting glass (0.0025 mm<sup>2</sup> / 0.04 mm<sup>2</sup>, Bürker CE, Germany) to count spores under microscope (Olympus BX 51). The spore concentration was determined by the following formula:

$$Spore Concentration = \left[ \frac{No. \ of \ spores \ in \ a \ 0.25 \ mm \times 0.25 \ mm \ square}{0.0000125 \ ml} \right]$$
(1)

The spore concentration was maintained at  $2 \times 10^6$  spores/ml. Fungal spore suspension was poured into an Erlenmeyer flask fitted with a glass nozzle and air pump. Wooden specimens were completely immersed in water for 2 days before inoculation. Five replicates of wood (70 mm x 70 mm) were spray-coated homogeneously by each type of fungal spore suspensions. Inoculated wood specimens were stored in glass covered plastic trays at a saturated humidity condition and ambient laboratory temperature (21  $^{\circ}C - 23 \,^{\circ}C$ ). The wood specimens in the trays were checked regularly to observe the growth of fungi. Each plastic tray contained water to maintain the atmospheric humidity at saturation level.

**Microscopy:** Substrate surface images were obtained by Olympus BX 51 microscope (4X magnification).

**FTIR spectroscopy**: The fungi grown on wooden surface and on the coating were harvested by spatula. Spectroscopic analysis was performed by an attenuated total reflection (ATR) - Fourier transform infrared (FTIR) spectrometer (Thermo Nicolet 8700) that was continuously purged with purge gas. Fungi specimens were pressed against a diamond crystal in the ATR Smart Orbit accessory of the FTIR instrument. An average of 32 scans at a resolution of 4 cm<sup>-1</sup> was recorded for each spectrum by the OMNIC software. No further spectral correction was performed.

### **RESULTS AND DISCUSSION**

The microscopic images shown in Fig. 1a and Fig. 1b prove that fungi grow on coated wood surfaces. The mycelium was observed to be concentrated at the corners of the coated wood specimens by naked human eye. However, microscopic evaluation proved that the whole coated surface got covered with shiny spider-web like mycelium threads that were more conglomerated at the edges possibly due to better connection with the wood that served as nutrient source.



Fig. 1a. Red coated surface.



Fig. 1b. Red coating fungal growth.

Fig. 2 shows the FTIR spectra of fungi that were cultivated on wood and fungi that were cultivated on wood coating. There are IR peaks at ~ 3280 cm<sup>-1</sup> for bonded O-H stretching vibration, ~2929 cm<sup>-1</sup> for fatty acids, ~ 1630 cm<sup>-1</sup> for C=O stretching vibrations of protein amide I, 1545 cm<sup>-1</sup> for in-plane bending of protein amide II, 1245 cm<sup>-1</sup> and 1030 cm<sup>-1</sup> for  $-CH_2OH$  stretching vibrations of carbohydrates. Magnified images of Fig. 2 are provided in Fig. 3- Fig. 6 for better evaluation.



Fig. 2. FTIR spectra of fungi grown on wood and wood coating.

Fig. 3 shows the representative spectra of fungi at the IR region of fatty acid groups. Fig. 4 shows the IR region of carbonyl groups. The spectra of fungi grown on coating and fungi grown on wood have similar qualitative characteristics without any dissimilar undulations. However, it was observed visible that the absorbance intensities varied quantitatively between specimens.



**Fig. 3.** FTIR spectra showing fatty acid region of wood fungi.

**Fig. 4.** FTIR spectra showing carbonyl region of wood mould fungi.

Fig. 5 shows the absorbance region of the cellulosic C-H at 898 cm<sup>-1</sup>. The fungi spectra that were grown on coating do not show any characteristic absorption at that wavenumber. The spectra from fungi that were grown on bare wood surface, however, show visible absorption at 898 cm<sup>-1</sup>. This indicates that the fungi that were grown on coating were starved off cellulosic nutrient source while the fungi that were grown on wood had natural source of nutrients. To be particular, these results stated that the fungi cell becomes what type of nutrition it obtains from substrates. More importantly, this finding tells that the wood surface coating obviously blocks the micro-organism to get access of the substrate part.



**Fig. 5.** FTIR spectra showing cellulose absorbance region of wood mould fungi.

**Fig. 6.** FTIR spectra showing the absorbance of C-H groups of wood mould fungi.

Fig. 6 shows the absorption region of C-H groups that occur at lower FTIR wavenumbers. There are not many differences between the spectra from fungi obtained from two nutrient types, except at the wavenumber  $872 \text{ cm}^{-1}$ . Fungi from coated surface

shows a peak in this FTIR region which is not so prominent from the spectra collected from fungi grown on wood.

### CONCLUSION

Mould fungus, *A. pullulans*, was cultivated successfully on coated surfaces of wood along with uncoated surfaces of wood. FTIR spectra of the harvested fungi were obtained by the application of the attenuated total reflectance (ATR) technique. It was found that the FTIR spectra of fungi obtained from two nutrient types, show similar pattern at wavenumbers between 4000-1000 cm<sup>-1</sup>. At lower wavenumbers, however, it was observed that the fungi grown on coating lacks the signature peak of cellulosic C-H group and show extra peaks that are absent in the fungi grown on wood. Henceforth, it can be concluded that ATR-FTIR may represent a useful tool to discover the nutrient sources of wood mould fungi.

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# EFFECTS OF WOOD PROPERTIES ON SURFACEMOULD GROWTH ON COATED CLADDINGS OF NORWAY SPRUCE

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### ABSTRACT

Development of surface moulds and staining fungi on painted spruce panels with known origin and wood properties was investigated over a period of 4 years. Materials of Norway spruce (Picea abies) were sampled from two sites with high-productive forest on lowland in South-eastern Norway and two low-productive sites at higher altitudes and somewhat farther north. Claddings were processed from inner centreboards (mainly heartwood) and outer centerboards of both butt logs and second logs. A sub-sample of radially sawn claddings was compared with corresponding tangentially sawn claddings. Heartwood proportion, density, annual ring width, knot diameters and knot area were measured. All panels were coated with the same water-borne alkyd modified acrylic paint system. Most of the tangentially sawn claddings were coated on the side facing pith, but a sub-sample was coated on the opposite side for comparison. The specimens were exposed with 45° angle of inclination facing south in a field trial in Oslo from 2007 to 2011, and mould growth was evaluated visually according to EN 927-3. 7.7% of the specimens were rated as 2, 71.4% were rated as 3, 19.4% were rated as 4, and 1.5% were rated as 5. Outer boards were rated significantly higher than inner boards, while differences between origins were not significant. There was a tendency of decreased rating with increasing heartwood proportion, but the relationship was not significant. Nor was there any significant effect of annual ring with, density or knot properties. Neither the difference between radially and tangentially sawn claddings, nor the difference between specimens coated on the side facing pith was significant.

Key words: coated cladding, moulds, Norway spruce, staining fungi, wood properties.

### INTRODUCTION

Wooden façades have traditionally been used in family houses in Norway and are increasingly applied also in multi storage and non-residential buildings. Norway spruce (*Picea abies*) is the most common wood species used for cladding material in façades. The house owners and other end-user demand cladding material with long maintenance

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intervals and long service life. Growth of mould fungi with dark coloured hyphea and spores are a common phenomenon on both painted and unpainted wooden façades, and will reduce the aesthetical service life of the facade. UV-radiation, rain, temperature, condensation, wind and high relative humidity degrade the surface of both painted and unpainted cladding and makes it more susceptible to fungal attack (de Meijer 2001). The chemical composition and the physical condition of the paint, together with the underlying wood substrate, are important factors for how the cladding performs (Ahola 1991, Richter et al. 1995, Williams et al. 2000). The overall effect will vary depending on the end-use of the material and the local climate (Brischke et al. 2006, Gobakken et al. 2008). Numerous studies have been performed on wooden claddings, and some have taken wood quality into consideration. Studies have been performed on the influence of surface roughness and porosity on coating adhesion (Nussbaum et al. 1998, Williams and Feist 1994), the influence of annual ring width on water sorption properties of wood (Flæte and Alfredsen 2004), and the influence of heartwood content on water sorption properties and fungal growth (Bergström and Blom 2005). In a study by Gobakken & Lebow (2010) it was found clear differences in degree of mould growth between various wood species when coated, and that there were indications that coated sapwood had more surface mould growth than coated heartwood. The degree of natural resistance to fungal growth in wood may vary considerably between species and stem positions. Heartwood in several species contains extractives with antifungal properties (Zabel and Morrell 1992).

The influence of wood-coating interactions on the growth of moulds and staining fungi is still not well enough known. In many studies of coated wood, the description of the wood is sparse, and the variation within the wood specimens seems to be of less interest. The objective of this study was therefore to analyze the effects of selected wood properties of Norway spruce on surface moulds and staining fungi when cladding panels were exposed outdoors.

### MATERIAL AND METHODS

In order to have different levels of annual ring width, materials of Norway spruce (Picea abies) were sampled from two fertile sites and two poor sites in South-eastern Norway. Sites were classified as poor if the site index, defined as dominant height at 40 years age, was 14 m or lower and as fertile if the site index was 20 m or higher. While the trees at the fertile sites (Larvik) were about 50 years old, had large annual ring width and small taper, the trees from the poor sites (Toten) were about 150 years old, had small annual ring width and larger taper. Five trees with breast height diameter between 27 and 30 cm and five trees with breast height diameter between 32 and 35 cm were sampled from each site. This sampling also represents some difference in wood density since it is negatively correlated to annual ring width within a site index and a geographical area (Klem 1934). Butt logs and second logs were processed to claddings with dimensions 19 x 98 mm<sup>2</sup>. While butt logs contain wood with small and dry knots, second logs usually contain wood with larger and often sound knots. The claddings to be tested were chosen from both inner centreboards and outer centreboards. The sawing pattern is presented in Fig. 1. Inner centreboards are mainly heartwood and contain some proportion of juvenile wood. Outer centreboards contain mature wood with longer fibres and smaller variation in wood density, and they may have some proportion of sapwood. A part of the material was produced as radially sawn claddings as seen in Fig. 1 (3), and those were compared to a corresponding part of tangentially sawn claddings.

A sub-sample of the panels was painted on the external face (the side facing the bark) to be compared to panels painted on the internal face (the side facing the pith). The samples represent a wide range of wood properties. Since the number of samples was limited, many properties were measured but not stratified in the sample such as heartwood proportion, annual ring width, density and knot size. Heartwood content was measures from CT-images of the timber scanned before drying. Knots were measured on the samples before painting. Annual ring width and density were measured from small pieces of clear wood.



**Fig. 1.** Claddings were produced from (1) inner centreboards, (2) outer centreboards, and from (3) radially sawn boards.

The samples were applied with one layer of a fully pigmented waterborne alkyd emulsion primer and two layers of a waterborne acrylic topcoat on the top face and the sides. The primer and the topcoat were applied by spraying in an automatic industrially setup. The panels were installed vertically at an angle of  $45^{\circ}$  in rigs facing south in Sørkedalen, Norway and were exposed from June 2007 until June 2011. During that period the panels were evaluated 7 times and the evaluations were performed in May/June and September each year. The mould coverage was evaluated according to EN 927-3 (2000). The assessment was made visually and by the use of a stereo microscope (x10 magnification) following a rating system with a range from 0 (no growth) to 5 (heavy growth) given by the pictorial rating scale in the EN 927-3 standard (2000). The rating is based on a step-wise increase in mould growth coverage, but the shape and the pattern of the mould growth are also of importance to the determination. The statistical calculations were performed in JMP (SAS Institute Inc 2010).

### RESULTS

In June 2011 the specimens had mould ratings between 2 and 5, with a majority of rating 3. 7.7% of the specimens were rated as 2, 71.4% were rated as 3, 19.4% were rated as 4, and 1.5% were rated as 5. As the exposure time progressed, an effect within the test site was detected. All specimens with rating 5 and 42% of those with rating 4 were located on a rack standing close to the edge of a forest. It was apparent that this location had an effect on the growth of fungi. If specimens on this rack were excluded, 8.6% of those remaining were rated as 2, 78.7% were rated as 3, and 12.7 were rated as 4. There also seemed to be an effect of location on the specimens with rating 2, of which 52% were located on a rack at the highest elevation of the test site. If also specimens located on this rack were excluded, 4.4% of those remaining were rated as 2, 81.9% were rated as 3, and 13.7% were rated as 4.

The statistical analyses were performed only on boards from butt logs since almost all the specimens on the two racks with apparent effects of location were from second logs. There was a tendency of higher rating in specimens with smaller heartwood proportion, but the effect was not significant, neither when heartwood was calculated as proportion of cross section ( $\chi^2$ =2.50, p=0.11) nor when it was calculated as proportion of the surface ( $\chi^2$ =1.99, p=0.16). The rating was not significantly influenced by annual ring width (( $\chi^2$ =61, p=0.43), density (( $\chi^2$ =2.01, p=0.16), maximum knot diameter ( $\chi^2$ =1.79, p=0.18), or with knot area ( $\chi^2$ =0.28, p=0.59). The difference between origins was not significant ( $\chi^2$ =0.98, p=0.61). There was a tendency of more rating 4 in specimens from Larvik than from Toten, but the difference was counterbalanced by a slightly higher proportion of rating 2 in specimens from Larvik. There was significantly higher rating in outer boards than in inner ( $\chi^2$ =6.27, p=0.04). The proportions of rating 3 were similar for inner and outer boards, but none of the outer boards were rated as 2. As compared with inner boards where both rating 2 and rating 4 appeared, more of the outer boards were rated as 4. The rating was not significantly different between specimens coated on the side facing pith and corresponding specimens painted on the side facing bark  $(\chi^2=2.39, p=0.30)$ , but the tendency was towards a higher rating on sides facing the bark. There was also a tendency of higher rating in specimens with radial surfaces than in corresponding outer boards with tangential surfaces, but the difference was not significant ( $\chi^2$ =1.40, p=0.24).

### DISCUSSION

Since wood is a heterogenous and non-isotropic material, variation in the wood properties will always be present. This might influence the effects of any treatments if it is not taken into consideration. According to Kollmann & Côtè (1968), the properties that influence most on sorption behavior are fibre angle, density and content of extractives, i.e. heartwood content. Williams et al. (2000) mentioned knots as an important factor for coating performance. In spruce, heartwood content has been found to influence growth of surface fungi and water affinity rather than water absorption (Bergström and Blom 2005).

In this study only centreboard selection was found to have a significant effect on surface moulds and staining fungi when Norway spruce claddings were exposed outdoors. Outer centreboards were found to have significantly higher mould rating than inner centreboards. Outer centreboards give a flat-grained cladding board. Moisture-induced deformations of wood might lead to cracks in coatings that are exposed to variations in temperature and relative humidity. Since shrinking in tangential direction is about twice as large as that in radial direction (Kollmann and Cöté 1968), one may expect more cracks in the coating and then a shorter lifetime for tangentially sawn claddings (flatgrain boards) cut from the outer centreboard than claddings from vertical- or edge-grain boards. A slightly more weathered paint film with cracks on claddings made from outer centreboard may give that extra adaption in micro climate for mould and staining fungi to grow better. In addition the outer centreboards have none or very little heartwood, and any antifungal effect from extractives was not present. The tendency of higher mould rating in specimens with smaller heartwood proportion and higher mould rating on sides facing bark support the results for the effect of centreboard selection. Radial surfaces swell and shrink less than tangential surfaces, and unexpectedly claddings with radial surfaces had the tendency of having higher mould rating than in corresponding outer boards with tangential surfaces.

The variability in rating was small apart from the specimens on the racks with apparent effects of location. Claddings with the highest mould rating were found on a rack standing close to the edge of a forest, where the trees may have caused a more sheltered environment (higher temperature, less wind and higher relative humidity). Also organic residues may have been deposited on the surface of the cladding giving enhanced growth conditions for mould and staining fungi. The elevation within the test site and the positioning of some surrounding trees may have caused a high portion of rating 2 on a rack at the highest elevation. Shorter periods of fog and more hours of direct sunlight may have resulted in less time-of-wetness and lower mould rating. Very small changes in temperature, wood moisture content and surface nutrients, which are the predominant factors for fungal growth, can have big impact on the onset fungal colonisation. The critical in-situ condition (CIC) for wooden components is exemplified by Gobakken et al. (2008) and shows the importance of parameters that serve as triggering factors for fungal growth for a specific component and how they can out rule other important properties.

Gobakken and Lebow (2010) reported from a field test of 4.5 years that wood substrate was significant but of less importance than coating and exposure time. Type, amount and composition of fungicides in the paint have a direct effect on the colonisation of mould fungi on the surface (Gobakken and Jenssen 2007, Van Acker et al. 1998, Viitanen and Ahola 1997) and may preclude any enhanced effect of the wood substrate.

More restrictions concerning the composition and the use of fungicides in coating emphasise the importance of more knowledge about how paint systems with less or without fungicide perform on different wood substrates. This study on coated claddings of Norway spruce showed that few of the studied wood properties affected the growth of moulds and staining fungi. But to be able to optimizing each step in the production of environmentally friendly, durable and aesthetical acceptable wooden façades for the future, more knowledge about wood properties in combinations with surface finishes and various climatic conditions are needed.

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# ABSORPTION OF LIQUID WATER IN SPECIMENS OF COATED CLADDING OF NORWAY SPRUCE (*PICEA ABIES* [L.] KARST.)

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### ABSTRACT

Liquid water absorption in small specimens of acrylic coated cladding was investigated before and after one year of outdoor exposure, and before and after 3000 h of exposure in a QUV chamber. Regarding the specimens exposed outdoors, water absorption was measured until more than 3000 hours, and desorption was measured as well. Statistical analysis was performed on amount of absorbed water after 72 and 672 h. Regarding the QUV exposed specimens, water absorption was measured after 72 hours only. In both experiments the specimens absorbed less water after exposure. Origin had effect in both experiments; samples from high productive sites with young trees had a higher water uptake than those from low productive sites with old trees. Horizontal orientation of the specimens (inner/outer board) had influence on the results both before and after outdoor exposure, but this effect was not found in the QUV exposed specimens. Regarding wood properties, no significant effect of density or annual increment width was found. In specimens exposed outdoors, heartwood content had large influence on water uptake. Outdoor exposure apparently had a smaller effect on water absorption in outer boards than in inner boards, causing the difference between outer and inner boards to be smaller after exposure.

Key words: acrylic coating, natural weathering, QUV, service life, water uptake

#### INTRODUCTION

The ability to withstand changes in moisture content is one of the most important properties of wooden claddings. One of the reasons for this is that changes in moisture content below fiber saturation leads to dimensional changes, which eventually leads to cracking of the wood. Another reason is that wood is vulnerable to biological deterioration when its moisture content approaches fibre saturation (Eaton and Hale 1993). Wooden claddings are often coated, both due to aesthetic preferences and in order to protect the wood from water and UV radiation (de Meijer 2001). The performance of the cladding is strongly influenced by the chemical composition and the

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physical condition of the paint, as well as the properties of wood substrate (Ahola 1991; Williams, Jourdain et al. 2000).

Water absorption in wooden claddings has been studied extensively, and some of the studies have taken wood quality into consideration. Among the various objects of study are the influence of annual ring width on water sorption properties in Norway spruce wood (Flaete and Alfredsen 2004) and the influence of heartwood content on water sorption properties in uncoated Norway spruce wood (Sandberg 2002; Bergström and Blom 2006).

Partly because of its low permeability, Norway spruce (*Picea abies* (L.)) is the most commonly used cladding material in Norway (Øvrum 2002). Trees grown in different conditions can have large differences in wood properties, which can be expected to have effect on water uptake. In an earlier publication regarding uncoated specimens from the same project as the present study, significant differences in water uptake were found between sample groups from different growth conditions and different horizontal locations in the log (Sivertsen and Vestøl 2010). Coatings provide barrier against water uptake. Application of a high- or medium-build coating has been found to reduce the effect of wood properties significantly (Jämsä and Viitaniemi 2006).

The objective of this study was to study the water uptake in coated wood with special emphasize on comparing results of artificial UV exposure with outdoor exposure, and to test the effects of selected wood properties.

# MATERIAL AND METHODS

Norway spruce material was sampled from two sites with high productivity (Larvik) and two with low productivity (Toten) in South-eastern Norway. The trees from the high productivity sites were about 50 years old, had large annual ring width and small taper. The trees from the low productivity sites were about 150 years old, had smaller annual ring width, and larger taper. Five trees with breast height diameter between 27 and 30 cm and five trees with breast height diameter between 32 and 35 cm were sampled from each site. Cladding material measuring 19 mm x 98 mm was produced from the butt log and the second log in each tree, with different horizontal positions (Fig. 1). This was done to ensure variation within each tree, as butt logs and second logs can be expected to contain wood with largely different knot patterns. Inner centreboards are mainly heartwood and contain some juvenile wood (Fig. 1, board 1). Outer centreboards contain mature wood with longer fibres and smaller variation in wood density, and they may contain some sapwood (Fig. 1, board 2). A part of the material was produced as quarter-sawn claddings (Fig. 1, board 3). The samples represent a wide range of wood properties. Since the number of samples was limited, many properties were measured but not stratified in the sample such as heartwood proportion, annual ring width, density and knot size. Heartwood content was measures from CT-images of the timber scanned before drying. The CT measurements were not performed on quarter-sawn boards or boards coated on the bark side. Knots were measured on the samples before painting. Annual ring width and density were measured from small pieces of clear wood.



**Fig. 1.** Boards were sawn from second logs (A and B) and butt logs (C and D) of trees with two different diameter classes. Board 1 was coated on the pith side (m) and on the bark side (y), board 2 on the pith side (l) and board 3 on either of the two radial faces (s).

The cladding boards were applied with one layer of a fully pigmented waterborne alkyd emulsion primer and two layers of a waterborne acrylic topcoat on the top face and the sides. The primer and the topcoat were applied by spraying in an automatic industrial setup. Most specimens were coated on the side facing pith (m), but a sub-sample was painted on the side facing the bark (y). The quarter-sawn claddings were coated on either of the two radial faces (s).

Water absorption was investigated according to EN 927-5, with certain modifications. The test faces were sawn and not planed, and the specimen size was not exactly as described in the standard. Due to restrictions in the QUV test setup, there was a difference in specimen size between this test and the outdoor exposure test. Specimens for outdoor exposure measured 70 x 98 mm (1 x b), although some boards had smaller breadth due to limitations from the diameters of the trees. Specimens for the QUV test measured 150 x 70 mm (1 x b). The thickness was 19 mm for all specimens.

In the outdoor exposure test, material from first and second logs, and inner (m) and outer (l) centreboards coated on the pith side, as well as radially sawn boards (s), were used. After the first water uptake test, the specimens were conditioned to 12 % MC, exposed on racks at a  $45^{\circ}$  angle towards the south for one year, and then tested again. With a total of 1008 h, the water uptake test was considerably longer than defined in the EN 927-5. Data from 72 and 672 hours of exposure were used in statistical analyses.

In the UV exposure test, only material from butt logs was used. Inner centreboards coated on the side facing pith (m), inner centreboards coated on the side facing bark (y), outer centreboards coated on the side facing pith (l) and radially sawn (s) boards were used. The UV exposure was done in a QUV test chamber, but with no spraying cycle. The duration of the water uptake test was 72 h, as defined in EN927-5.

The statistical calculations were performed in JMP (SAS Institute Inc 2010).

### RESULTS

The water uptake was higher before exposure than after exposure in both parts of the experiment. The water uptake in the specimens studied in the outdoor exposure experiment was slightly higher than in the QUV exposed specimens if only comparable specimens were included (Table 1). Before exposure, more water was absorbed by the specimens in the outdoor exposure specimens than in the UV exposure specimens. After exposure, the specimens from both exposures absorbed similar amounts of water.

**Table 1.** Water absorbed during 72 h floating before and after outdoor exposure andQUV exposure respectively.

Exposure	Water absorption during 72 h floating (g/m <sup>2</sup> )				
	Least Sq Mean <sup>*</sup>	Std Error			
Before outdoor exp.	649 A	11.6			
Before QUV	470 B	14.6			
After outdoor exp.	265 C	11.6			
After QUV	224 C	14.6			

<sup>\*</sup> Different letters behind the values mean significant difference in Tukey Kramer comparison.

#### **QUV** exposed specimens

The water uptake was significantly higher in specimens from the high productive sites (origin Larvik) than in the specimens from the low productive sites (origin Toten). Radially sawn claddings absorbed significantly more water than the outer centreboards, but the difference in absorbed amount was small. Tree size, density and year increment width were not significant.

#### **Outdoor exposed specimens**

The difference in water uptake between exposed and unexposed specimens was more pronounced in the beginning of the experiment (Fig. 2). After 336 hours the mean curves of the two groups showed a parallel increase.



O — Mean g/m2 coated exposed + — Mean g/m2 coated unexposed

**Fig. 2.** Mean absorption curves (absorbed water, weight per area of the exposed face) for all specimens before and after exposure.

The water uptake was significantly higher in specimens from the high productive sites (Larvik) than from the low productive sites (Toten), and it was also significantly higher in specimens from the outer centreboards (l) than those from inner centreboards (m). Radially sawn (s) specimens did not have significantly different water uptake than inner centreboard (m) specimens or outer centreboards (l) specimens. The difference between inner and outer centreboards was explained by heartwood proportion, but origin still had significant effect that could not be explained by the recorded wood properties.

#### DISCUSSION

The two different exposure tests gave similar changes in water uptake before and after weathering. This indicates that both short-term outdoor exposure and UV exposure can be used to assess development in water uptake in coated wood with ageing.

The permeability of acrylic coatings is known to decrease with leaching and short-term ageing (De Meijer and Nienhuis 2006). The fact that this development was found in both the outdoor exposure and the UV exposure seems to indicate that this is mainly due to the effect of UV radiation, as no spraying was performed during the UV exposure. However, due to the large temperature variations there will always be condensation on the specimens during parts of the exposure cycle. Based on earlier experience, the amount of water generated by this condensation is probably enough to cause some leaking of coating components (pers. comm. Guido Horn, WKI, Germany).

The results from the outdoor exposure test show that heartwood content was the most important of the recorded wood properties. The variation in heartwood content in the specimens used in the UV exposure test was too small to justify statistical testing. The effect of origin is significant in both experiments, indicating influence from site effects that have not been taken into account in the models. Possible effects could be grain angle or proportion of bordered pits in ray tracheids (Liese and Bauch 1967; de Meijer, Thurich et al. 1998).

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# CARBON FOOTPRINT OF WOOD PRODUCTS – METHODOLOGICAL ISSUES AND SOME EXAMPLES FROM NORWAY

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### ABSTRACT

Greenhouse gas (GHG) emissions due to human activities have increased since preindustrial times. Climate change arising from anthropogenic activity has been identified as one of the greatest challenges facing countries, governments, business and people with major implications for both human and natural systems. Several initiatives are being developed and implemented to limit GHG concentrations in the atmosphere. Such GHG initiatives rely on the assessment, monitoring, reporting and verification of GHG emissions and/or removals.

Goods and services account for a major share in overall GHG emissions. Gaining a better understanding of these emissions as well as pathways for their reduction is of prime importance, in the supply chain as well as with consumers. GHGs are emitted, stored and removed throughout the life cycle of a product (i.e. cradle-to-grave) from raw material acquisition through production, use, and end-of-life treatment. A carbon footprint of a product measures the product's life cycle GHG emissions. In this paper we discuss relevant methodological issues as well as previous and ongoing work in Norway on carbon footprints of wood products.

Key words: Carbon footprint, greenhouse gases, wood products, life cycle assessment

### INTRODUCTION

Greenhouse gas (GHG) emissions due to human activities have increased since preindustrial times, with an increase of 70% between 1970 and 2004. Carbon dioxide (CO<sub>2</sub>) is the most important anthropogenic GHG. Annual CO<sub>2</sub> emissions grew by about 80% between 1970 and 2004. Global atmospheric concentrations of CO<sub>2</sub>, methane (CH<sub>4</sub>) and nitrous oxide (N<sub>2</sub>O) have increased markedly since 1750 and now far exceed pre-industrial values determined from ice cores spanning many thousands of years (IPCC 2007).

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Climate change arising from anthropogenic activity has been identified as one of the greatest challenges facing countries, governments, business and people with major implications for both human and natural systems. Several initiatives are being developed and implemented to limit GHG concentrations in the atmosphere. Such GHG initiatives rely on the assessment, monitoring, reporting and verification of GHG emissions and/or removals.

Goods and services and their consumption account for a major share in overall anthropogenic GHG emissions. Gaining a better understanding of these emissions as well as pathways for their reduction is of prime importance, in the supply chain as well as with consumers. GHGs are emitted, stored and removed throughout the life cycle of a product (i.e. cradle-to-grave) from raw material acquisition through production, use, and end-of-life treatment. In recent years, the carbon footprint has gained recognition as an indicator of the contribution of goods and services to climate change. It is often based on a life cycle approach.

A carbon footprint of a product measures the product's life cycle GHG emissions. In this paper we discuss relevant methodological issues and previous and ongoing work in Norway on carbon footprints of wood products.

# CARBON FOOTPRINT METHODOLOGY

Numerous climate calculators, e.g. for calculating a person's carbon footprint, have been developed around the world. Many of the tools can be useful for reducing GHG emissions. However, in many cases these tools suffer from not being transparent in describing how the carbon footprints actually are calculated. It is therefore difficult to evaluate if all relevant elements are included in the calculations.

PAS 2050 (PAS 2050 2008) is a publicly available specification (PAS) prepared by BSI, the national standards body of the UK, to specify requirements for assessing the life cycle GHG emissions of goods and services. PAS 2050 was published in 2008 and is currently under revision.

Another example of carbon footprint systems is The Greenhouse Protocol developed by the World Resources Institute (WRI) and the World Business Council for Sustainable Development (WBCSD). The GHG Protocol comprises two separate but linked specifications, one for accounting and reporting GHG emissions of companies/organisations, and one for quantifying reductions from GHG mitigation projects. WRI and WBCSD are planning to publish a protocol for products in 2011.

There are also ISO standards that are relevant for carbon footprint assessment (Table 1). A carbon footprint can be quantified either at the product or service level as in Life Cycle Assessment (LCA) described by ISO standards 14040 and 14044 (2006) or at an organisation or company level as described in ISO standard 14064-1 (2006).

Standard	Title					
ISO 14020: 2000	Environmental labels and declarations — General principles					
ISO 14021: 1999	Environmental labels and declarations — Self-declared					
	environmental claims (Type II environmental labelling)					
ISO 14024: 1999	Environmental labels and declarations — Type I environmental					
	labelling — Principles and procedures					
ISO 14025: 2006	Environmental labels and declarations —Type III environmental					
	declarations — Principles and procedures					
ISO 14040: 2006	Environmental management — Life cycle assessment —					
	Principles and framework					
ISO 14044: 2006	Environmental management — Life cycle assessment —					
	Requirements and guidelines					
ISO 14064-1:	Greenhouse gases — Part 1: Specification with guidance at the					
2006	organization level for quantification and reporting of greenhouse					
	gas emissions and removals					
ISO 14064-2:	Greenhouse gases — Part 2: Specification with guidance at the					
2006	project level for quantification, monitoring and reporting of					
	greenhouse gas emission reductions or removal enhancements					
ISO 14064-3:	Greenhouse gases — Part 3: Specification with guidance for the					
2006 validation and verification of greenhouse gas assertions						
ISO 14065: 2007	Greenhouse gases — Requirements for greenhouse gas validation					
	and verification bodies for use in accreditation or other forms of					
	recognition					
ISO 14066: 2011	Greenhouse gases — Competence requirements for greenhouse					
	gas validation teams and verification teams					

Table 1. ISO-standards related to carbon footprinting.

Presently, there is on-going work in the International Organization for Standardization (ISO) to develop an international standard for quantification and communication of carbon footprints of products (ISO 14067). The standard is expected to be released in 2012. In the draft issue of ISO 14067 the quantification of GHG emissions and removals of a product shall follow a life cycle approach, and it complies with the ISO 14040-series (LCA) and ISO 14025 (Type III environmental declarations).

According to ISO CD3 14067 a carbon footprint of a product is the "sum of GHG emissions and GHG removals of a product system, expressed in CO<sub>2</sub> equivalents" based on a life cycle approach. Relevant GHGs are carbon dioxide (CO<sub>2</sub>), methane (CH<sub>4</sub>), sulphur hexafluoride nitrous oxide  $(N_2O)$ ,  $(SF_6),$ hydrofluorocarbons and perfluorocarbons. The CO<sub>2</sub> equivalent of a specific amount of a GHG is calculated as the mass of a given GHG multiplied by its global warming potential (GWP). GWP is a relative measure of how much heat a GHG traps in the atmosphere. It compares the amount of heat trapped by a certain mass of the GHG to the amount of heat trapped by the same mass of carbon dioxide. A GWP is calculated over a specific time interval, commonly 100 years, but other time intervals like 20 or 500 years can be used. The Intergovernmental Panel on Climate Change (IPCC) provides updated GWPs of different GHGs (IPCC 2011).

# CARBON FOOTPRINTS OF NORWEGIAN WOOD PRODUCTS BASED ON ISO 14025 (TYPE III ENVIRONMENTAL DECLARATIONS)

Table 2 shows  $GWP_{100}$  in kg  $CO_2$  equivalents per functional unit for various Norwegian wood products. The figures are collected from Environmental Product Declarations (EPDs) produced according to ISO 14025. The EPDs are available at www.epd-norge.no.

Table 2.  $GWP_{100}$  (kg CO<sub>2</sub>-eqv) for wood products from Norwegian EPDs. FU: Functional Unit, ESP: Expected Service Life.

Product	FU	<b>GWP</b> <sub>100</sub>	Scope of assessment	ESL	EPD No.
Dried sawn timber	$1 \text{ m}^3$	19.1	Cradle to gate	-	NEPD
					nr: 082E
Planed structural timber	$1 \text{ m}^3$	28.9	Cradle to grave	60	NEPD
					nr: 084E
Copper-impregnated timber,	$1 \text{ m}^3$	33.2	Cradle to gate	-	NEPD
NWPC class AB					nr: 087E
Exterior cladding treated	$1 \text{ m}^2$	5.6	Cradle to grave	50	NEPD
with waterborne acrylic paint					nr: 137E
Gluelam	$1 \text{ m}^3$	78.8	Cradle to grave	60	NEPD
					nr: 115E
Cross laminated Timber	$1 \text{ m}^3$	102.6	Cradle to grave	60	NEPD
(CLT)					nr: 114E
Interior wood cladding,	$1 \text{ m}^2$	0.4	Cradle to grave	30	NEPD
untreated					nr: 085E
Exterior cladding treated	$1 \text{ m}^2$	5.6	Cradle to grave	50	NEPD
with waterborne acrylic paint					nr: 137E
I-beam, H: 400 mm	1 m	1.7	Cradle to grave	60	NEPD
					nr: 088E
Iso3 stud, 47 x 200 mm (load	1 m	1.9	Cradle to grave	60	NEPD
bearing wooden stud isolated					nr: 124E
with polyurethane)					

As shown in Table 2 the functional unit are not the same for all products. By converting the untreated interior wood cladding and the exterior cladding treated with waterborne acrylic paint to 1 m<sup>3</sup> the corresponding GWP<sub>100</sub> are 24.7 kg CO<sub>2</sub>-eqv and 200.7 kg CO<sub>2</sub>-eqv respectively. The overall picture is that additives, such as glue and surface treatment are important contributions to the GWP<sub>100</sub> of wood products.

A product influences climate at many stages from raw material acquisition through production, use, and end-of-life treatment. This requires a holistic environmental assessment and LCA is regarded to be a well suited tool for this purpose. However, it is impossible to cover all methodological issues for all possible product groups in one common standard. This is where Product Category Rules (PCR) come into use. For all products listed in Table 2 a PCR developed for solid wood products were used (NPCR 015 2009).

The PCR specifies requirements for collection of data and how to perform calculations for a product group. A PCR document defines functional unit, system boundaries, cut off rules, allocation rules, etc., and enable transparency and comparability between different products within a product group.

Many of the processes in the forest products industries produce multiple outputs, and it is therefore necessary to allocate impacts onto each output. This requires a systematic assessment of the material flows. For many products the carbon footprint is exclusively related to emissions of fossil GHGs. However, many products comprise biogenic carbon and/or bioenergy is utilized for manufacturing of the products. This is the case for wood products. It is not yet clear how this should be handled. The biogenic carbon in the wood is originating from  $CO_2$  captured from the atmosphere photosynthesis of a tree and stored as lignocelluloses.

Following NPCR 015 (2009) emissions or removals of  $CO_2$  from biogenic carbon should not be included because combustion of wood fuels is considered equal to the  $CO_2$  uptake in the forestry growth process. However, in ISO CD3 14067 GHG emissions and removals arising from fossil carbon sources and biogenic carbon sources and sinks shall be included in the inventory and shall be documented separately in the carbon footprint study report. How emissions/removals arising from biogenic carbon sources are taken into account in a carbon footprint is not defined.

The above mentioned methodological issues are not only important for comparison of different products. It is also crucial when analysing how the forest resources best can be utilised in mitigating climate change. This is one of the main objectives in the ongoing Norwegian research project ClimateWood (www.klimatre.no)

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# A SITUATIONAL ANALYSIS OF THE FURNITURE MANUFACTURING INDUSTRY IN LATVIA

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### ABSTRACT

The furniture manufacturing industry is an important, innovative and environmentally friendly industry that the turnover is more billions of euros per year and it consists of small and medium-sized enterprises. Latvian forest industry is oriented to quality and high value-added wood products productions and delivery to the consumer. In Latvia, the wood and wood products are increasing every year. However, the production of high value-added wood products is too low.

The study objective was to investigate and obtain the updated information on the furniture industry enterprises activity in the Latvia, their products and potential for the future. To achieve this objective was necessary to obtain the information on the types of products, its quality and the distribution channels, the markets, the enterprises dispersal and customer structure. In the study the respondents interviewing method was used and the target group is the Latvia's furniture enterprises.

The study showed that the Latvian furniture industry is characterized by regional dispersion and it is the large number of micro, small and medium-sized enterprises. According to research results, the main manufactured products are the cabinet furniture, cafe, office and hotel furniture, children furniture, solid wood, exclusive furniture and wooden windows, doors and stairs. Most manufactured products the quality level judged as high or very high, except with certain exceptions. The furniture enterprises manufactured products marketed primarily on the domestic market. The main enterprises customers are the private individuals and juridical persons and most effective way of acquisition customer is the existing and new customers' recommendations.

Key words: furniture manufacturing industry, forest enterprises, Latvia

### INTRODUCTION

The furniture manufacturing industry is essentially an assembling industry, which employs various raw materials to manufacture its products. They range from wood and wood based panels to metals, plastics, textile, leather and glass. There are many different types of furniture (e.g. chairs, sofas, tables, wardrobes, kitchens, mattresses) with very different uses (e.g. households, schools, offices). The European furniture sector comprises around 150,000 companies, generates a turnover of almost  $\in 126$  billion and an added value of  $\in 38$  billion and employs around 1.4 million people (EU27, 2006) (The furniture industry..., 2011).

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Furniture-making is a sector which has long-standing and stable traditions in Latvia, although the types of furniture that are manufactured have changed over the course time. Until the early 1990s, most furniture manufactured in Latvia was made of wood chip plates. Since the restoration of Latvia's independence, high value furniture, includes solid timber furniture, has become the main type of product (Forest sector..., 2008).

In 2008 compared to 2007, the consumption and exports of furniture decreased and the furniture consumption on the domestic market was LVL 87 million (EUR 124 million) and exports was only LVL 66 million (EUR 94 million). However, the last two years have been a very bad, because the domestic furniture consumption was only LVL 42 million (EUR 60 million) in 2009 and in the first half of 2010, but the export of furniture was LVL 54 million (EUR 77 million) and LVL 52 million (EUR 74 million) (Kas notiek..., 2010). The Latvian furniture industry is the potential to develop in the future.

### MATERIALS AND METHODS

The study target group is the Latvia's furniture enterprises. The planned sample size was 778 respondents, where had submitted their annual report for the year 2009 in the Latvian companies public database "Lursoft". The achieved sample size was 604 respondents. The study was conducted in early 2011. In the study, the respondent interviewing method with telephone interviews was used that to ascertain the information to the pre-prepared questions.

For data processing the statistics treatment methods are used: descriptive statistics and data intercomparison. Since this is the first study on the furniture enterprises with such a large number of respondents. The study results can be used as the reference for other studies, the mutual data comparison and evaluation.

The study results are always with a certain degree of statistical error probability. It has been taken into account whilst analyzing and interpreting the study results. If these differences are included in the statistical error limits or less, then they can be regarded as minor. The statistical error is calculated by the following formula (Meža nozares..., 2009):

 $SE = q \times \overline{\pi} \times (100 - \pi)/n$ 

where:

SE – statistical error;

q – the coefficient that at 95% probability is

equal to 1.96;

 $\pi$  – in the study achieved responses of the

percentage breakdown;

n – the number of respondents.

To determine the statistical measurement error, unweight number of respondents in the group and the results as a percentage are given. Using these values, the section can find the measurement error limits of +/- percentage with 95% probability.

#### **RESULTS AND DISCUSSION**

According to the obtained results of the study, 41% (318 respondents) of all respondents confirmed and 7% (56 respondents) of all furniture enterprises refused the participate in this study. However, during financial crisis 21% (159 respondents) of all respondents changed their working area and 15% (118 respondents) of all enterprises ceased their

activities or went into liquidation or declared insolvency proceedings. Approximately 15% (118 respondents) of all respondents were unable to find the contact information or phone numbers was closed, it could not obtained the information about these companies. Only 1% (9 respondents) of all questionnaires was filled in part. For this study, in 2010 the furniture manufacturing industry heavily influenced the financial and economic crisis that contributed the "weak" players missing, allowing other furniture enterprises to more easily overcome this crisis.

According to the obtained results of the study from 313 respondents showed that 79% of all enterprises was the joinery enterprises and only 21% was the industrial manufacturers, which demonstrated that Latvian furniture manufacturing industry was more oriented to the individual orders, not for on mass production in large quantities.

#### **Regional dispersion and employment**

In Latvia are five region and they are Kurzeme, Zemgale, Vidzeme, Latgale and Riga and its region. The study showed that in the furniture manufacturing industry is characterized the regional dispersion. The largest volume of enterprises located in Riga and its regions, it is nearly 66% of all respondents, but 34% located in other Latvian region. According to the obtained results of the study, 10% furniture enterprises located in Latgale region, leaving behind the Vidzeme region with 9%, Zemgale region with 8% and Kurzeme region with 7%. Top three cities, where loceted the most furniture enterprises, is Riga, Daugavpils and Jelgava. In Riga located 42% enterprises of all respondents, but in Daugavpils and Jelgava located 5% and 4% furniture companies. In Daugavpils and Jelgava the furniture manufacturing industry is the traditional sector and historically in these cities were the major furniture factory and after their disposal remained the lot of specialists who could started own businesses.

The obtained results of the study showed that in the Latvian furniture manufacturing industry dominated the micro enterprises, which employed up to 9 employees and is 69% of respondents. The second most important group is small furniture enterprises which employed from 10 to 49 employees and its volume is 25% of all respondents. However, the third most important group is the medium-sized enterprises, which employed from 50 to 249 employees and its volume is only 6%. According to the obtained results, the large furniture enterprises are not in Latvia (Fig. 1.).



Fig. 1. Breakdown by enterprises groups, %.

According to the obtained results of the study, in all Latvian regions, including Riga regions, dominated the micro-enterprises. The most micro-enterprises are in Latgale regions, forming 75% of all respondents. In second place is Riga and its regions with 71% and third place – Zemgale regions with 67% (Fig. 2.).

The obtained results showed that the second important group is the small furniture enterprises and in all Latvian regions the proportion is more or less the same, i.e.  $\sim 25\%$  of all respondents. However, the number of medium-sized furniture enterprises is

different in Latvian regions, for example, in Kurzeme regions 33% of all respondents are the medium-sized furniture enterprises, but in Latgale regions are not the medium-sized furniture enterprises (Fig. 2.).



According to the obtained results, in Latvian furniture manufacturing industry most people employed in Riga and its regions, i.e. 49% of all respondents. The second most important regions are Vidzeme with 21%, then Zemgale with 13%, Kurzeme with 10% and Latgale with 7%.

#### Production

The obtained results of the study showed that in Latvia the furniture enterprises most produced the kitchen furniture, bedroom furniture and office furniture (Fig. 3.).



Fig. 3. Enterprises produced furniture, the number of respondents.

Latvian furniture enterprises produced the products after individual orders and not showing the products saving, thereby are able a more flexibly responded to changes in market and better meet the customer needs. However, under the section "other furniture" are included the enterprises, which mainly produced the stairs and doors, wooden windows, wooden chests, cabinet, chidren, soft and solid timber furniture, as well as other wood products and its components.

According to obtained results of the study, 89% of all respondents claimed that their manufactured products quality level is the high and very high, of which 22% of all respondents confirmed that their product quality level is very high, but 67% - - high. However, only 11% of all respondents claimed that the products quality level is medium. None of the respondents their products quality level was not classified as medium-low or low.

The obtained results showed that the main products consumer group is the private persons, which approved 226 surveyed enterprises. As the second most important group
is the juridical persons, which marked 186 furniture enterprises of all respondents. The third important consumer group is the state and local authorities (35 enterprises of all respondents).

Latvian furniture manufacturing enterprises the main distribution channel of products is the consumer recommendations or "word of mouth" dissemination of information and 250 enterprises marked it. Only 15 enterprises of all respondents used own catalog to distributed the products and 5 enterprises doing through its supplier catalog (Fig. 4.).



Fig. 4. Distribution channels of products, the number of respondents.

During a more detailed analysis of the other distribution channels, the results showed that a very popular way how to disseminate the information about the products is the dissemination of information on the internet and the homepage. The part of respondents claimed that the information about the products disseminated through own or the broker shops and salons, designers, architects and builders, as well as through the advertising and informational brochures.

According to the obtained results of the study, Latvian furniture enterprises the manufactured products mostly realized on the domestic market and 56% of all respondents confirmed that works only in the Latvian markets, bet 44% of respondents claimed that have founded the export markets for their products. The results of the study showed that 47% of the enterprises exported their products to 25%, but 25% of all respondents – from 26 to 50%. However, 28% of all respondents claimed that product export is over 50% of the output, of which 19% of the enterprises said that product exports is over 75%.

According to the obtained results, in furniture manufacturing industry is very closely related to the markets of furniture fittings, because often directly the furniture fittings increased the product price and quality. The study showed that the key furniture fitting enterprises in Latvia. The most commonly cited (185 respondents) the furniture fitting enterprise is SIA "AM Furnitūra" (Fig. 5.).



**Fig. 5.** Key furniture fittings enterprises in Latvian furniture manufacturing industry, the number of respondents.

In the second place is SIA "Daiļrades Koks" (134 respondents) and third place – SIA "Attēls R" (99 respondents). As major furniture fitting suppliers in Latvia are mentioned as follows enterprises - SIA "Hetla", SIA "Furlats", SIA "Roja", SIA "Quadrex", SIA "SB Partneri", SIA "Baija" un SIA "Būvlukss". The obtained information showed that overall are many furniture fittings suppliers in Latvia.

Currently, there is a relatively low demand for an exclusive design or solid wood furniture, this is due to market recovery after the crisis, but it is only a matter of time when the Latvian people will resume in the solvency of stabilization. Can predict the industry's output demand increases, especially after natural wood furniture, as demand for organic products in Europe is currently at a high level, as well as Latvian is a trend to promote eco-lifestyles, which can be a challenge for furniture enterprises to produce on the basis on how to combine natural materials with style elements.

## CONCLUSIONS

1. In the furniture manufacturing industry is characterized the regional dispersion an the largest volume of enterprises located in Riga and its regions, it is nearly 66% of all respondents, but 34% located in other Latvian region. In the industry dominated the micro enterprises, which employed up to 9 employees and is 69% of all respondents. Latvian furniture enterprises most produced the kitchen furniture, bedroom furniture and office furniture. In the Latvian furniture manufacturing industry 89% of all respondents claimed that their manufactured products quality level is the very high and high. The main products consumer group is the private persons and as the second most important group are the juridical persons. Latvian furniture manufacturing enterprises the main distribution channel of products is the consumer recommendations. Latvian furniture enterprises the manufactured products mostly realized on the domestic market (56% of all respondents), but 44% of respondents claimed that have founded the export markets for their products. In the furniture manufacturing industry is very closely related to the markets of furniture fittings, because often directly the furniture fittings increased the product price and quality.

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# NATURAL BUILDING MATERIALS: WHAT ARE THE USERS' PERCEPTION OF NATURALNESS?

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## ABSTRACT

Visual representations of nature, natural objects and design elements, symbols of nature etc. can evoke positive experiences in the built environment. Over the past decades, a number of empirical studies have documented that both passive and active experiences of nature may be beneficial for human health and well-being, for example reduced stress levels and increased well-being and positive emotions and feelings.

In order to exploit the possible beneficial effects of natural building materials, there is a need for knowledge about which materials users perceive as natural. The present study investigates people's perception of naturalness for various building materials. The study includes material samples from solid wood products, processed wood, engineered wood products, wood-based fibreboards, surface treated wood, masonry, wallpaper, tiles, metal and plastic. The study concluded that some materials such as wood and stone are perceived as natural. The degree of transformation that the material undergoes during the production process influences the perceived naturalness.

Key words: Natural materials, focus group, building material

## INTRODUCTION

Naturalness is a preferred characteristic of most materials. Evidence for this have been provided for range of product groups, such as foodstuffs and medicines (Rozin 2004) and construction materials from wood (Overvliet and Soto-Faraco 2010). Research conducted on foodstuffs imply that naturalness of a particular substance is connected to the history of transformation it has undergone from its original state: contagion, chemical changes, processing and mixing (Rozin 2004).

The aim of the study is to 1) investigate what makes people perceive a material as natural or the opposite and 2) evaluate how people perceive various building materials with respect the characteristic of naturalness. The present study is motivated by results from environmental psychology and evidence based design.

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### METHOD

The study is conducted in two stages. First, a focus group analysis was conducted in order to explore the concept of "naturalness" (Norwegian: *naturlighet*) and to identify building materials that are of relevance in the interior environment and material use. Second, a survey was conducted where respondents evaluated various material samples.

#### Focus group analysis

Focus group analysis is a qualitative research method and is usually conducted as an explorative study. Four focus group discussion were organized, 28 persons participated in the focus group anlaysis (Nyrud et. al 2010). All participants were recruited in the Oslo-region, and were between 24 and 40 years old. The focus groups were structured according to ethnic background (one group consisted of persons of persons who had grown up outside Scandinavia), experience with construction or remodeling and gender.

The group discussions were conducted in accordance with a prepared interview guide and were moderated by a researcher. All focus group discussions were transcribed based a sound recording of each discussion.

#### Survey

Respondents were recruited from a student association and two athletic associations. They were asked to evaluate various building materials on a binomial scale, i.e. state if they perceived the material as *natural*, a negative answer implies that the material was considered *not natural*. The material samples measured 100 mm x 100 mm. All samples were presented in close fitted cardboard box and the participants were allowed to evaluate the samples both visually and tactilely. The respondents were free to evaluate the samples the any order they preferred. The material samples are described in Table 3.

Almost 90 % of the respondents were 35 years or younger (cf. Table 2), about 65 % of the respondents were female (cf. Table 3).

Table 1. Ag	ge distribution among	Table 2	. Gender distribution.
re	espondents.		
Age interval	Number of respondents	Gender	Number of respondents
15-25	32	Female	37
16-35	18	Mala	10
36-45	4	Wale	19
46-55	1		
56-65	1		

## RESULTS

Results from the focus groups indicates that the main criteria for characterizing a material as natural, was that it does not contain chemical compounds and/or has not undergone significant transformation. This corresponds well with previous research reported by Rozin (2004). The use of wood as a building material is largely considered to be something positive – especially when used as flooring. The materials must, however, correspond to the environment they are used in. Variation and contrasts in the use of materials has a positive influence on indoor environment.

Results from the survey indicates that some materials are unambiguously perceived as either natural or not natural. Some materials are, however, receiving ambiguous results. The number of respondents that rated the products as natural are displayed in Table 4 and the relative share of the respondents rating materials as natural are presented in Figure 1.

Two types of materials are considered natural; wood and stone. The untreated, uncanted pine (344) was rated as natural by all respondents. Untreated, planed, pine with knot (113) and untreated, planed, pine clearwood (420) as well as untreated natural stone tiles (510) were all rated as natural by more than 95 % of the respondents.

Among the materials rated as not being natural are, as expected metal, plastics and woven fabric. There are no respondents that have rated the white painted steel (773) as natural. Untreated steel (447), plastic (642) and wowen fabric made from untreated sacing (307) are rated not natural by approximately 95 % of the respondents.

Material	Tree species	Sample number	Natural	Not natural
Pine, untreated, sawn, uncanted	Pinus silvestris	344	56	0
Pine, untreated, planed, with knot	Pinus silvestris	113	55	1
Pine, untreated, planed, clearwood	Pinus silvestris	420	54	2
Natural stone tiles, untreated	Fraxinus exelsior	510	54	2
Ash, untreated, heartwood	Pinus silvestris	615	49	7
OSB, untreated		007	44	12
Brick tiles, untreated		235	41	15
Cork		193	35	21
MDF (12 mm), untreated		712	31	25
Particleboard, untreated		158	28	28
Pine, surface trated, planed, white Wowen fabric, wool		560 469	28 26	28 30
MDF (imitated year rings), surface treated, white		401	24	32
Leather, untreated		829	17	39
Wallpaper, white		823	16	40
MDF (12 mm), surface treated, white		210	14	42
Wood-plastic composite, imitated year rings		321	13	43
Cheramic tiles, glazed, white		292	11	45
Wowen fabric, untreated sacing		307	5	51
Plastic, untreated, polished		042	4	52 52
Steel, untreated, milled surface		44 /	3	55
Steel, surface treated, white		773	0	56

**Table 3.** Number of respondents ratings materials as natural/not natural.



Fig. 1. Relative share of respondents rating the materials as natural (dark grey) not natural (light grey).

## DISCUSSION AND CONCLUSIONS

The materials characterized as most natural were wood and stone, whereas the materials characterized as not being natural were steel, plastic and woven fabric. The results correspond well with the results from previous research. There appears to be a distinction between materials that have undergone little transformation in the production process (solid wood and stone) and materials that have undergone a substantial transoformation (metal or plastics). In the case of wood products, most sawn wood samples are considered natural by more than 95 % of the respondents and about 90 % perceive OSB as natural, but wood based panels made from smaller fragments, such as MDF and particleboard, are considered natural by only 50 % of the respondents.

There is also a distinction between materials that have been treated with chemical compounds, for example paint or other surface treatments. Untreated wood is considered natural by practically all respondents, whereas a layer of surface treatment will make 50 % of the respondents consider the material not natural.

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# ACTIVITY-BASED COSTING IN WOOD VALUE CHAIN: SAWMILLING CASE

Korpunen, H.<sup>1</sup> & Uusitalo, J.<sup>2</sup>

## ABSTRACT

Production cost controlling is one important strategic task in any branch of industry, in sawmilling as well. Some of the production costs are also manageable elements in price setting. There is a need of precise knowledge of how the costs really act when some factor of production changes. This study tests an activity-based costing (ABC) method for a large scale sawmill that refines approximately 350 000 m<sup>3</sup> roundwood annually. Sensitivity and applicability of the costing model were analysed with two case studies. This study supports the fact that the ABC method is a useful tool in cost controlling at a sawmill; and a cost structure of a sawmill has an effect on the logistic chain of timber procurement.

Key words: Activity-based costing, sawmilling, sawing pattern, profitability

### INTRODUCTION

The value chain of wood-based products starts in the forest. Forest machine operator makes tree bucking decisions according to the demand and pricing information received from wood conversion factories. The demand and price matrices that guide the logging are eventually based on the market information of finished products: the products that are desired in markets will have an emphasis on the demand and price matrices. Unbiased pricing of the products is thus in a crucial position because the decisions in the forest are irreversible. Open markets set the prices of the products, which means that the industry must follow and control its own costs.

Many managerial accounting systems are initially developed for factory level cost calculations, but in the forest industry, the mill level information is not accurate enough. When the costs must be allocated to individual product groups, activity-based costing (ABC) method is useful. The ABC was developed to provide more accurate information of profitability of the production (Turney, 2005). The basic principle of the ABC is that the production costs in each process are allocated to products by consumption of resources. The ABC method serves sawmilling excellently, since the production is clearly process-based and raw materials and finished products may each vary significantly.

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The aim of the study was to evaluate applicability of the activity-based costing method in sawmilling industry. The system analysis tested the effect of two different sawing patterns to a softwood sawmill production costs and the profits (later referred as Case 1 and Case 2). The results of the study can be further adapted in raw material allocation to different wood end users.

## MATERIAL AND METHODS

The material was collected in 2009–2010. The production of a virtual Scots pine sawmill was handled in eight processes (Table 1.). This process definition was based on several interviews with sawmill managers. The production costs were allocated with a cost driver, which was lead-time of each product. Machinery and other resource costs and technical information were collected from international sawmill machinery manufacturers, other professionals and statistics, the information is presented in more detail in Korpunen et al (2010).

**Table 1.** Production processes of a industrial-level softwood sawmill.

Receive, unload and sorting of logs	Debarking	Sawing and edging	Green sorting and stickering	Drying	Quality sorting and packing	Storing and shipping	Chip and sawdust production
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The key variable in the study was the sawing pattern. In the CASE 1 we had normal sawing pattern with 6–8 lumber pieces in a log class. In CASE 2 we reduced the amount of sideboards to 4–6 lumber pieces in a class. The sawing patters are presented in Table 2. Tested sawing patterns were adapted from Uusitalos (1995) research.

## **Table 2.** Sawing patterns for CASE 1 and CASE 2. Length-% describes the relationbetween the log and the board.

CASE	1
------	---

Small end		Cente	erpieces					Boa	ards			
diameter	Thickn.	Width			Thickn.	Width			Thickn.	Width		
class (cm)	(mm)	(mm)	Lenght-%	Pcs.	(mm)	(mm)	Lenght-%	Pcs.	(mm)	(mm)	Lenght-%	Pcs.
17	50	100	100	2	19	100	90	4				
19	50	125	100	2	19	100	95	4				
21	50	150	95	2	19	100	90	6				
23	50	150	100	2	19	125	95	2	19	100	95	4

CASE 2

Small end		Cente	erpieces					Boa	ards			
diameter class (cm)	Thickn.	Width (mm)	Lenght-%	Pcs.	Thickn.	Width (mm)	Lenght-%	Pcs.	Thickn.	Width (mm)	Lenght-%	Pcs.
17	50	100	100	2	19	100	90	2	(11411)	()	Long.u /o	1 001
19	50	125	100	2	19	100	95	2				
21	50	150	95	2	19	100	90	4				
23	50	150	100	2	19	125	95	2	19	100	95	2

We used a log distribution which consists of approximately 346 000  $\text{m}^3$ -solid logs. The logs were sawn by the sawing patterns of CASE 1 and CASE 2. The volumes of each log and end product classes are presented in Table 3.

Log length class (m)	Small end diameter class (cm)	Logs	Log volume (m <sup>3</sup> )	Lumber v	olume (m <sup>3</sup> )	Chips and volum	d sawdust e (m <sup>3</sup> )	Bark volume (m <sup>3</sup> )
				CASE 1	CASE 2	CASE 1	CASE 2	
	17	120000	14304	8083	6442	3761	5402	2460
4.0	19	120000	17520	9466	7733	5146	6879	2908
4,0	21	120000	21024	11765	10123	5643	7285	3616
	23	120000	24960	12832	11099	8035	9768	4093
	17	120000	15377	8689	6925	4043	5807	2645
10	19	120000	18834	10176	8313	5532	7395	3126
4,3	21	120000	22601	12647	10882	6066	7831	3887
	23	120000	26832	13794	11931	8638	10500	4400
	17	120000	16450	9296	7408	4325	6212	2829
16	19	120000	20148	10885	8893	5918	7911	3345
4,0	21	120000	24178	13530	11642	6490	8377	4159
	23	120000	28704	14756	12764	9240	11233	4707
	17	120000	17522	9902	7891	4607	6618	3014
4.0	19	120000	21462	11595	9473	6304	8427	3563
4,9	21	120000	25754	14412	12401	6913	8924	4430
	23	120000	30576	15719	13596	9843	11966	5014

**Table 3.** Distribution of input logs, by log, lumber, chips & sawdust and bark volumes.For CASE 1 and CASE 2.

For the profit calculation a price list of different product groups was also generated, the price list is presented in Table 4. We used an average lumber price for all lumber classes.

Table 4. Price of different product groups at mill gate.

Log price at mill gate,	Lumber price,	Chips and sawdust price, €/m <sup>3</sup> (solid)	Bark price, €/m <sup>3</sup>
$\notin/m^3$	€/m <sup>3</sup>		(solid)
65	150	20	6

The net profit of product group i (groups are bark, lumber, chips and sawdust) was calculated with following formula (Equation 1).

$$Net_profit_i =$$
 (price - production\_cost) volume Eq.1

Total profit calculation for sawmill was done with Equation 2.

$$Mill \_ profit = raw \_ material \_ \cos t - \sum_{i} Net \_ profit_{i}$$
 Eq.2

### RESULTS

Processing costs are presented for each end product class by each process and then summed for total production costs. Total costs of production in CASE 1 and CASE 2 are presented in Table 5.

								1	
	Receive			Green		Quality	Storing	Chips and	
CASE 1	unload &		Sawing and	sorting and		sorting &	and	sawdust	
	log sorting	Debarking	edging	stickering	Drying	packing	shipping	production	Total
Bark	88703	47370							136073
Chips and sawdust	153181	81803	300946					210974	746904
Lumber	285851	152653	561596	716268	2951263	892795	512540		6072966
CASE 2									
Bark	88703	47370							136073
Chips and sawdust	198955	106248	390876					210974	907053
Lumber	240077	128208	471667	708875	2647794	876037	512540		5585198

**Table 5.** Production costs (€) of CASE 1 and CASE 2 in each process.

In CASE 1, the unit production costs of bark were 2.34  $\notin$ /m<sup>3</sup>, chips and sawdust 7.43  $\notin$ /m<sup>3</sup>, lumber 32.38  $\notin$ /m<sup>3</sup>. In CASE 2 the production unit costs were: bark 2.34  $\notin$ /m<sup>3</sup>, chips and sawdust 6.95  $\notin$ /m<sup>3</sup>, lumber 35.46  $\notin$ /m<sup>3</sup>.

The energy intensive drying process caused most of the annual costs, depending on the sawn lumber dimensions it generated 40% of the costs. Production costs of chips and sawdust was 10–13% of the annual production costs.

With product group prices, costs and volumes, profit calculation returned +1029231 € for CASE 1. The profit of CASE 2 was -2547329 €.

## DISCUSSION AND CONCLUSIONS

The comparison between CASE 1 and CASE 2 revealed that when the amount of boards decreased, total production costs followed. At the same time, the unit costs of lumber production increased, this was mainly due to lower utilization rate of the production. Reason for cheaper production of chips and sawdust was basically derived from the same origin; the machinery was used more efficiently. The cost of processing bark remained the same in both cases because the changes in sawing pattern did not change the amount of bark and the same amount of resources was still needed for debarking the logs. Direct continuum to this thinking is that the lumber yield should be minimized in order to get the lowest costs. This leads to point where all logs are chipped and no lumber is produced anymore. This thinking was proven to be false; the profits gained from different end products directed the production, not merely the production costs. It is also notable that the technical limitations must to be taken into account when estimating the best possible production pallet.

As the cost of raw material remained the same in the profitability calculations, and the net profit of the most important product group (lumber) was decreased by over 4 million

euros from CASE 1 to CASE 2, the increment in volume of chips and sawdust was not sufficient enough to keep the mill profitable in CASE 2.

As a result, a mill manager has to be able to control the costs and production planning in order to get the highest value from the raw material. Mere cost reduction is not necessarily the key in achieving the most profitable production. This study gives some new ideas for supply chain management and new orientation for estimating the most profitable wood conversion chains.

Activity-based costing was proven to be an effective tool for cost controlling. Processbased thinking helps tracing weak spots in production and the improvements are easier to focus on right processes. The results of the study indicated that the sawing pattern has a clear effect on the sawmilling costs and profits. As the sawing pattern is formed according to market situation and the properties of logs, and the properties of logs are determined in the forest level, straight interaction between forest machines and sawmill is economically beneficial for the entire value chain.

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# MATERIAL FLOWS IN THE NORWEGIAN SAWMILLING INDUSTRY

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## ABSTRACT

In Norway, the forest resources in the standing forest and the annual harvest of industrial roundwood are well monitored. However, the flows and stocks of wood resources throughout the industrial value chains have no coherent information. The purpose of this work is to quantify the main flows of wood resources into and out of the Norwegian sawmilling industry. Material flow analysis (MFA) is the main method applied with a combination of statistical data and process parameters. MFA is a method for analyzing the flows and stocks of materials and substances through a well defined system. It is based on the law for conservation of mass, i.e. that the inputs of a process must equal the outputs plus the change in the stocks of the process. The system boundaries used are the Norwegian sawmilling industry and the foreign markets of its products. Production data are gathered from the sawmilling industry's trade association and national industry and trade statistics. Average process parameters are estimated from data gathered in previous studies. The results provides a holistic quantification of the internal and external utilization of the different wood products.

Key words: Material flow analysis, sawmilling industry, resource availability.

### INTRODUCTION

The sawmilling industry is driven by its main product, sawn timber, but it also produces several co-products. Many of the co-products have changed from being unwanted waste to demanded products in several other industries.

There have been several previous studies on the material flows in the Norwegian forest based industries. Rødland (2009) has mapped the wood flow from the forest based value chains from stump to end use in Norway for the year 2006. Grinde (2010) used these data to build physical make-use tables for the forest based industries in Norway. Both Rødland and Grinde pointed out the lack of previous work and challenges in data availability on the material flows in the forest based industries. In the context of estimating the amounts of carbon stored in wood products, Gjesdal et al. (1998) have estimated the flows of carbon from forestry and net import through the industrial sectors to storage in products and to municipal waste in 1993.

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There have been several studies conducting surveys in the Norwegian Sawmilling industry. Tengs (1994) have gathered volume balances for nine sawmills in Norway, but experienced substantial uncertainties in amounts of wastes and co-products. Building on this work, the data basis was improved and used to make environmental product declarations (EPD) for Nordic wood products (Tengs and Opdal, 1997). Wærp, Grini, Folvik and Svanæs (2009) have more recently made EPDs for the Norwegian wood products and a survey for production in 2007 was therefore conducted. Of the individual sawmills in Norway, Moelven Van Severen (2009) have an ISO 14001 environmental management system and as part of this publish material flow data annually.

The objective of this study was to quantify the main flows of wood in the Norwegian sawmilling industry.

### MATERIALS AND METHODS

Material flow analysis (MFA) is a method for analyzing the flows and stocks of materials and substances through a well defined system (Brunner and Rechberger 2004). It is based on the law for conservation of mass, which means that the inputs of a process must equal the outputs plus the change of stocks inside the process. System boundaries are the Norwegian sawmilling industry and the foreign trade markets of the main products. In MFA, it is useful to distinguish between transformation processes and market processes. Transformation processes are typical industrial processes and the parameter values are determined by the technology used. In market process on the other hand, the flows are set by prices controlled by the domestic and foreign supply and demand. Data material are collected from Statistics Norway, Trade Associations and from previous data survey at Norwegian Institute of Wood Technology. An overview of the material sources are shown in Table 1 bellow.

Process data	Sources	Activity data	Sources
Generation of co-	Tengs and Opdal	Domestic logging	Skog-data AS (2011)
products	(1997), Moelven Van	Bioenergy	Statistics Norway
	Severen (2009)	production	(2011a)
Wood densities	Bramming (2006)	Foreign trade	Statistics Norway
			(2011b)
Ash contents	Belbo and Gjølsø	Sawn wood	Treindustrien (2011)
	(2000)	production	

**Table 1.** Process and activity data with sources.

The system was design based on the flow diagram presented in Svanæs (2004) and illustrated using STAN (subSTance flow ANalysis), which is a software for material flow analysis. The advantage of using it is that the width of the flows visualize the amount of the flow, known as Stankey flows (IWA, 2011).

#### RESULTS

The results are presented in Fig. 1 and Fig. 2. Volume flows in solid cubic meters are shown in Fig. 1. One should keep in mind that flows upstream to debarking are counted

as over bark. The flows between the atmosphere and combustion are not counted in volumes as the low density of these flows would make to wide Stankey flows compared to the rest of the system. There is a total of 11 processes, which is divided into seven transformation processes and four market processes. The process saw wood storage, had a stock increase of about 12 % of the inflow. There are a total of 33 flows, where 18 are in or out of the system and 15 are within the system. Three are elementary flows to and from the environment, six are flows to the national economy and eight are flows of foreign trade. The last flow is balancing the volume loss from shrinking of wood during drying and is assumed to have no dry mass as seen in Fig 2.



**Fig. 1.** Volume flows in the Norwegian sawmilling industry and main markets in 2010.



**Fig. 2.** Dry mass flows in the Norwegian sawmilling industry and main markets in 2010.

## DISCUSSION

The results indicate that the consumed saw logs mainly comes from domestic sources. The bark, sawdust and hogs are partly being used within the industry for generation of thermal heat for drying the wood. The sawdust and wood chips are not only being utilized further domestically, a large share of these flows is either export or import. From the industry, the mass not yet being further utilized are ash from combustion that are landfilled and flue gas that are released to the atmosphere.

The total amount of bark inputs to the industry are found here to be substantially larger than in Rødland (2009). Considering common bark rates and the production of bioenergy in the sawmill industry, the amount of bark in Rødland is likely half of the actual amount. However, the ratio between internal use for bioenergy and external

supply to domestic market indicates that the use of bark in the industry has been reduced. Most like due to increased market price for bark and hence improvements in energy efficiency.

Andersson and Opdal (1996) found that the production of 1 sm<sup>3</sup> of sawnwood generates 1.5-2.5 kg of ash. In 2010. 2 115 000 sm<sup>3</sup> sawnwood was produced, this would result in between 3172.5 and 5287.5 ton of ash.

The foreign trade data from Statistics Norway are report in value, weight and volume. Calculations on densities for chips and sawdust, indicate that these varied with years and for import and export at a range of 135-918 kg per cubic meter. Therefore it is likely that there are large uncertainties in the foreign trade data. This is probably due to variation in use of solid and lose cubic meters as unit.

## CONCLUSIONS

- First coherent study of material flows for entire sawmilling industry
- Uncertainties are large in foreign trade statistics and likely cause is mixed use of units in reporting.
- Most outflows of the industry are sold as products.
- Potential for further utilization of outflows are ash and flue gas.
- Further studies should also include planning and preservation of wood in the industry and include carbon footprint, energy and value added.

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## WOOD IN URBAN BUILDING CONSTRUCTION: A SURVEY OF NORWEGIAN ARCHITECTS AND ENGINEERS ATTITUDES

## Bysheim, K.<sup>1</sup> and Nyrud, A.Q.<sup>2</sup>

## ABSTRACT

Norwegian architects' and civil engineers' attitudes to using wood in major urban building constructions were investigated. Wood currently has a relatively small share of the market in urban construction, and increased use of wood as building material in urban building constructions is important for the wood industry. The principal objective was to develop knowledge about the mechanisms influencing key players when choosing building materials. Structural interviews (n=15) and a quantitative survey (n=203) was carried out to acquire further knowledge about the specifiers making the decisions regarding choice of building materials. The criteria investigated included attitudes towards the physical and mechanical properties of wood, perceived risk of using wood as a building material, and the environmental properties of wood. The knowledge status and use of information sources among the specifiers was also investigated. Factor analysis revealed previous experience, perceived fire related properties of wood and norms in the construction industry as important factors when using wood as a structural material. Perceived visual properties, fire related properties and previous experience were important factors when using wood as a facade material.

Key words: Theory of planned behavior, wood, structural material, façade material.

## INTRODUCTION

Today, the market share of timber in urban areas is low, particularly compared to construction materials such as concrete and brick. A better understanding of the mechanisms that influence key specifiers in their choice of building materials is needed, in order to understand how the industry can market wood as a construction material in urban areas. In this paper, the focus is on factors influencing architects' and structural engineers' use of wood as both a structural and a facade material in urban construction.

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## MATERIAL AND METHODS

Using theoretical models by Emmitt and Yeomans (2008) and Ajzen (1991), a model for the specifiers' decision making process was developed. Based on the model and previous research (Emmitt and Yeomans 2008, Bysheim and Nyrud 2009) an interview guide was developed. Six building projects in urban areas in Norway were chosen as case studies, and a total of 15 interviews were conducted among key players in the construction industry, such as engineers, architects and builders.

#### Structured interviews

The interviews were mainly focusing on three different main themes:

- 1. Knowledge and previous experience with wood as a building material
- 2. Important criteria for the selection of building materials
- 3. Role of decision makers and stakeholders in the building process

#### Survey

The results from the structural interviews, along with findings in previous research, formed the basis for a quantitative survey among key specifiers in the Norwegian construction sector. The criteria investigated included:

- 1. Attitudes towards the physical and mechanical properties of wood
- 2. Perceived risk of using wood as a building material
- 3. The environmental properties of wood
- 4. The knowledge status and use of information sources among the specifiers
- 5. Choice of building materials in the different stages of the building process

An invitation to a web-based questionnaire was sent by email to 2374 Norwegian architects, engineers, builders and contractors. This resulted in 203 answers, yielding a response rate of 8.6 percent. The educational background of the respondents were mainly architecture (N=139) and engineering (N=60). The questions in the questionnaire used a seven-point scale (1=to a small degree; 7=to a large degree). High value reflected a stronger preference for wood, except for items regarding risk and fire. Seven items were reverse-scored.

## RESULTS

#### **Factor analysis**

Exploratory factor analysis was performed to identify the least number of items to represent the interrelation between the variables. Fifty-four items from the questionnaire regarding criteria for the selection of wood materials were subjected to exploratory factor analysis with maximum likelihood extraction method. Separate analysis were

conducted for the two models, one for variables related to the use of wood as a structural material (SM) and one for the use of wood as a facade material (FM). The suitability of data for factor analysis was assessed. Inspection of the correlation matrix revealed the presence of a few loadings of .3 and above for both models. Factor analysis with oblimin and varimax rotations revealed very similar solutions. Bartlett's test of sphericity reached statistical significance (.000) for both models, supporting the factorability of the correlation matrix (Pallant, 2007). Inspection of the correlation matrix revealed the presence of loadings of .3 and above (table 1Feil! Fant ikke referansekilden. (SM) and table 2 (FM)). Factor analysis with oblimin and varimax rotations revealed very similar solutions. To aid in the interpretation of the components, oblimin rotation was performed. A minimum loading threshold of .3 was used. This revealed a simple structure (Pallant, 2007), with most components showing strong loadings, and all variables loading substantially on only one component. After inspecting the scree plot, examining the results of a parallel analysis and a test for very simple structure, it was decided to retain 11 (SM) and 10 factors (FM) for further analysis. Oblimin rotation with maximum likelihood extraction methods were used for both models. Total variance explained was 55.6% for the SM model, and 50.9% for the FM model.

#	Factor	Variance	Explanation
1	Visual	7.3%	Attractive appearance, vis. properties important for choice of
			building material, easy to combine with other mat., suitable for
			design tasks
2	Risk	6.4%	Increased risk of financial overruns, delays and building related
2	$C \rightarrow C$	C 10/	
3	<i>Combustible</i> ( <i>Fire</i> )	6.1%	Increased risk of damage in case of fire, exterior cladding
			increases risk of fire spreading, when used in load bearing
			structures it increases risk of fire
4	Environment	5.9%	Easily recyclable, env. friendly, low CO2 emissions, env.
			properties important for choice of building material
5	Experience (1)	5.1%	Experience with wood in apartment buildings, buildings for commercial purposes, public buildings, in interior and as
			facade material
6	Norm (1)	5.1%	Perceived attitude towards wood by builder, engineer and contractor
7	Perc heh control	4 2%	I am qualified to use wood wood is easy to use. I can use wood
,		1.270	if I want to
8	Experience (2)	4.1%	Experience with wood in primary construction and decking
9	Intentions	4.0%	I plan to use wood, I want to use wood as a structural material
10	Norm (2)	3.8%	Perceived attitude towards wood by real estate agent and public authorities
11	Prefab /calc	3.8%	Existing prefabricated solutions are satisfactory, access to wood design software is satisfactory

**Table 1.** Factor analysis: wood for structural purposes.

#	Factor	Variance	Explanation
1	Visual	6.8%	Attractive appearance, visual properties important for choice of building material, easy to combine with other materials, suitable for design tasks
2	Combustible (fire)	6.2%	Increased risk of damage in case of fire, exterior cladding increases risk of fire spreading, when used in load bearing structures it increases risk of fire
3	Risk	6.1%	Increased risk of financial overruns, delays and building related errors
4	Environmental	5.9%	Easily recyclable, env. friendly, low CO2 emissions, env. properties important for choice of building material, readily available material
5	Norm (1)	4.9%	Perceived norm towards wood by builder, architect, engineer and contractor
6	Experience	4.8%	Experience with wood in primary construction, non-bearing timber frame walls and decking
7	Norm (2)	4.6%	Perceived attitude towards use of wood as facade material by builder, architect, engineer, contractor
8	Intention	4.3%	I want to, I plan to use wood as a facade material
9	Norm (3)	3.7%	Perceived norm towards use of facade material by real estate agent, public authorities
10	Local sourcing/pre- fabrication/ software tools	3.4%	Local suppliers make wood an attractive material, Existing prefabricated solutions are satisfactory, access to wood design software is satisfactory

**Table 2**. Factor analysis: wood for facade purposes.

#### Anova analysis

Oneway between groups analysis of variance (Tukey HSD) was conducted to explore the impact of occupation and education on the aggregated variables used in the regression analysis. The subjects were divided into five groups according to occupation. Mean values for the aggregated variables are shown in table 3 and 4, were values not connected by same letter are significantly different. Three variables showed a statistically significant difference among the different occupations at the p <.05 level for wood as a structural material. Variable 1 (visual) indicated that architects (mean = 5,9) had a significant more positive attitude towards the visual properties of wood compared to engineers (4.9), builders (5.2) and contractors (4.8). For variable 8 (norm/PBC), there was a significant difference between architects (4.6) and engineers (4.0). This indicates that architects have a more positive perception of their own ability to use wood and how engineers and contractors will influence the choice of wood as a structural material. Architects (3.7) also had a significantly higher intention (variable 9) to use wood as a structural material than builders (2.5).

	Factor										
	1	2	3	4	5	6	7	8	9	10	11
Architect	5.9 <sup>A</sup>	5.4 <sup>A</sup>	4.1 <sup>A</sup>	5.8 <sup>A</sup>	6.0 <sup>A</sup>	4.4 <sup>A</sup>	2.0 <sup>A</sup>	4.6 <sup>A</sup>	3.7 <sup>A</sup>	3.9 <sup>A</sup>	3.4 <sup>A</sup>
Engineer	4.9 <sup>B</sup>	5.0 <sup>A</sup>	3.4 <sup>A</sup>	5.5 <sup>A</sup>	5.3 <sup>A</sup>	4.3 <sup>A</sup>	2.0 <sup>A</sup>	$4.0^{\mathrm{B}}$	3.1 <sup>AB</sup>	3.6 <sup>A</sup>	3.8 <sup>A</sup>
Builder	5.2 <sup>B</sup>	5.1 <sup>A</sup>	4.2 <sup>A</sup>	5.9 <sup>A</sup>	4.6 <sup>A</sup>	4.4 <sup>A</sup>	1.8 <sup>A</sup>	4.3 <sup>AB</sup>	2.5 <sup>B</sup>	4.1 <sup>A</sup>	3.7 <sup>A</sup>
Contractor	$4.8^{B}$	5.3 <sup>A</sup>	3.8 <sup>A</sup>	5.3 <sup>A</sup>	6.3 <sup>A</sup>	4.4 <sup>A</sup>	2.4 <sup>A</sup>	$4.6^{AB}$	3.3 <sup>AB</sup>	3.9 <sup>A</sup>	3.0 <sup>A</sup>
Other	5.8 <sup>AB</sup>	5.4 <sup>A</sup>	4.8 <sup>A</sup>	5.6 <sup>A</sup>	5.2 <sup>A</sup>	4.0 <sup>A</sup>	2.0 <sup>A</sup>	$4.4^{AB}$	3.2 <sup>AB</sup>	3.6 <sup>A</sup>	3.1 <sup>A</sup>

Table 3. Oneway Anova: wood as a structural material.

For wood as a facade material, there was a statistically significant difference (p < .05) for two variables: variable 1 (visual) and 8 (intentions). Architects (5.9) had a more positive attitude towards the visual properties of wood, same as the results presented in table 1. Architects (4.6) also had significantly higher intentions to use wood as a facade material than engineers (4.0).

	Factor									
	1	2	3	4	5	6	7	8	9	10
Architect	5.9 <sup>A</sup>	4.1 <sup>A</sup>	5.4 <sup>A</sup>	5.8 <sup>A</sup>	6.0 <sup>A</sup>	2.6 <sup>A</sup>	2.9 <sup>A</sup>	4.2 <sup>A</sup>	3.9 <sup>A</sup>	4.0 <sup>A</sup>
Engineer	$4.9^{\mathrm{B}}$	3.4 <sup>A</sup>	5.0 <sup>A</sup>	5.5 <sup>A</sup>	5.3 <sup>A</sup>	$2.7^{A}$	2.7 <sup>A</sup>	3.4 <sup>AB</sup>	3.6 <sup>A</sup>	$4.0^{A}$
Builder	5.2 <sup>B</sup>	4.0 <sup>A</sup>	5.0 <sup>A</sup>	5.9 <sup>A</sup>	4.6 <sup>A</sup>	2.2 <sup>A</sup>	3.1 <sup>A</sup>	3.2 <sup>B</sup>	4.1 <sup>A</sup>	4.2 <sup>A</sup>
Contractor	$4.8^{B}$	3.7 <sup>A</sup>	5.3 <sup>A</sup>	5.3 <sup>A</sup>	6.3 <sup>A</sup>	3.1 <sup>A</sup>	3.4 <sup>A</sup>	$4.0^{AB}$	3.9 <sup>A</sup>	3.7 <sup>A</sup>
Other	5.8 <sup>AB</sup>	4.6 <sup>A</sup>	5.4 <sup>A</sup>	5.6 <sup>A</sup>	5.2 <sup>A</sup>	2.4 <sup>A</sup>	2.9 <sup>A</sup>	3.3 <sup>AB</sup>	3.7 <sup>A</sup>	3.7 <sup>A</sup>

**Table 4.** Oneway Anova: wood as a facade material.

## DISCUSSION

The interpretation of the results from the structural interviews, the quantitative survey and the factor analysis was consistent with previous research (Emmitt and Yeomans 2008, Bysheim and Nyrud 2009, Wagner and Hansen 2004, Roos et al. 2010, O'Connor et al. 2004) suggesting experience, perceived risk, fire properties, visual properties and perceived behavioural control as important factors influencing the choice of wood as a structural material.

Factor analysis revealed that the four most important factors influencing the choice of wood as a structural and facade material were the visual properties of wood, perceived risk of using wood as a construction material and the combustive and environmental properties of wood. Perceived norm in the construction industry, previous experience and perceived behavioural control were also found to be important factors influencing choice of wood as building material.

Anova analysis revealed several statistically significantly differences among the specifiers, depending on occupational background. Architects had a significantly higher intention to use wood than the other major specifiers, both as a structural and facade material. Architects also had a more positive attitude to the visual properties of wood

than engineers, builders and contractors. The perceived behavioural control over the use of wood as a structural material was also higher among architects than engineers.

## CONCLUSION

Factors influencing choice of wood as a structural and facade material:

- Visual properties
- Perceived risk of using wood
- Combustible (fire) properties
- Environmental properties

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# THE WATER VAPOUR SORPTION PROPERTIES OF THE PLANT CELL WALL

## Hill, $C.A.S.^1$

## ABSTRACT

The water vapour sorption behaviour of several plant species has been studied. Results are discussed in terms of the equilibrium state (sorption isotherm, hysteresis) and also the kinetic behavior under conditions of adsorption and desorption. The results are considered by reference to the micromechanical behaviour of the cell wall matrix macromolecules. The implications for this work to further our understanding of the water vapour sorption properties of wood, plant fibres and other swelling gel systems are presented.

Key words: Water vapour, sorption, kinetics.

## INTRODUCTION

The relationship between atmospheric relative humidity (RH) and the equilibrium moisture content (EMC) of a material in contact with the atmosphere at constant temperature is known as a sorption isotherm. If material is exposed to an atmosphere containing water vapour it will increase in mass asymptotically until it eventually reaches the EMC, a process known as adsorption or absorption. Adsorption refers to a process of sorption onto a surface whereas absorption refers to sorption into the bulk of a material. The term adsorption is used throughout this paper to denote sorption onto the internal surface of the cell wall, since at a molecular level it is not really possible to differentiate a surface from the bulk. If a material has been maintained at a specific RH such that it achieves equilibrium and is then placed in an environment at a lower RH, it will lose water until it achieves a new EMC. It is well established that plant materials exhibit sigmoidal water vapour sorption isotherms and hysteresis between the adsorption and desorption branches of the isotherm, however a complete understanding of these phenomena is lacking. Plant-derived natural fibers (and wood!) absorb atmospheric moisture due to the presence of hydroxyl (OH) groups associated with the cell wall macromolecules. These can be broadly classed into three types. Cellulose which is the primary reinforcing element of the cell wall is made of linear chains of glucose residues which aggregate into microfibrillar units, possessing a high crystalline content (inaccessible to water molecules) and also a paracrystalline component to which water molecules can gain access, it thus has a high OH to C ratio but not all of the OH content is accessible. Lignin occurs in plant fibers to varying degrees, and is an amorphous crosslinked polymer composed of phenolic units and has a relatively low OH to C ratio compared with polysaccharides. There is also a hemicellulose and/or

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pectin component, which is predominantly an amorphous polysaccharide, is highly accessible to water molecules and has a high OH to C ratio. The cell wall of a plant microfibrils embedded fiber consists of crystalline in an amorphous lignin/hemicellulose/pectic matrix. As the cell wall absorbs moisture, the sorbed water molecules occupy space between the microfibrils resulting in expansion of the material. The expansion of the matrix requires work to be performed and this expansion will continue until the energy expended in expansion equals the free energy of the water vapour molecules in the atmosphere. At this point a state of equilibrium is obtained. The space that the water molecules occupy in the cell wall is referred to as the transient microcapillary network. Water within the cell wall is considered to be associated closely with the OH groups (monolayer water) or occurs within the transient microcapillaries but not intimately associated with OH groups (polylayer water). Although cell wall bound water can be classified in this way, this is not to suggest that the situation is static since there is continual exchange of molecules between the monolayer and polylayer. Concepts such as monolayer and polylayer are also of dubious value when considering the complex, dynamic geometry of the internal cell wall surface in any case. The existence of hysteresis means that the EMC values of the isotherm do not represent true equilibrium states and therefore should not be subjected to thermodynamic analyses since the states achieved are not path independent (this is often referred to as irreversible sorption). Thus, we are not in a position where we can confidently state that we understand water sorption in the cell wall. This paper presents some of the most recent research being performed at the Forest Products Research Institute at Edinburgh Napier University into the interesting and complex subject of water vapour sorption into the cell wall of plants.

### MATERIAL AND METHODS

The experimental details for these experiments have been described in considerable detail previously (Hill et al. 2009, Hill and Xie 2011). All data collected for this study utilised a Surface Measurement Systems (London) dynamic vapour sorption analyser. Sample masses were small (around 5 mg). Typical sorption experiments were programmed to run from 0 to 95% RH in 5% RH steps. The instrument is also programmed to maintain a constant RH state until the rate of mass change (dm/dt) reaches a specified value; this is to make sure that the experiments can be performed over a reasonable time scale. It has been found by trial and error that a dm/dt mass change of less that 0.002% over a ten minute period is suitable for obtaining EMC values within 0.1% of the values at infinite time. It is also possible to set the rate of data capture; in practice, it has been found satisfactory to record mass data every 20 seconds. The instrument also records real-time RH and temperature data with the sample mass information. Curve fitting to the kinetic data was performed using Origin 8.5 software (OriginLab Corporation, Northampton, MA, USA).

#### **RESULTS AND DISCUSSION**

#### **Sorption isotherms**

A comparison of the sorption isotherms of three plant fibres is shown in Fig. 1a, for Sitka spruce at different temperatures (Fig. 1b) and for Scots pine and thermally modified Scots pine (Fig. 1c) below. A number of interesting features are discerned in these plots. In Figure 1a it can be seen that coir has the highest sorption out of all of the materials studied and also has the greatest extent of hysteresis. Hemp has higher EMC values compared with cotton, but has a lower extent of hysteresis. In Fig. 1b isotherms are shown for Sitka spruce at temperatures ranging from 14°C to 44°C, as the temperature of the isotherm increases, the desorption curve shifts to the right, but there is no change in the adsorption curve, the net result is that the size of the hysteresis loop decreases. In Fig. 1c, there is a comparison of Scots pine and thermally modified Scots pine (3h at 200°C) it can be seen that although the thermally modified wood exhibits lower levels of sorption, there is a higher level of hysteresis compared with the unmodified wood.



**Fig. 1.** A comparison of the sorption isotherms of cotton, hemp and coir (a), Sitka spruce (b) and Scots pine (c).

It is interesting to note that coir, with the highest levels of lignin, also displays the highest levels of sorption - what does this tell us about sorption? It is quite clear that plant materials are hygroscopic because of the presence of hydroxyl (OH) groups, but it also appears that OH group concentration is not the sole determinant in controlling sorption behaviour. Of the plant fibres illustrated here, coir is the most elastic and exhibits very high strains to failure compared to many other plant fibres due (in part) to a high microfibril angle with respect to the long axis of the cell (Hill et al. 2009). The behaviour of Sitka spruce is interesting (Fig. 1b), in that the temperature affects the desorption branch of the isotherm but not the adsorption branch. Furthermore, the area enclosed by the hysteresis loops decreases as the isotherm temperature is increased. Another interesting aspect with respect to hysteresis is illustrated in Fig. 1c, which shows that the area enclosed by the hysteresis loop for thermally modified Scots pine is larger than the area bounded in the isotherm associated with the unmodified Scots pine. All of this behaviour can be explained by a model which considers the interaction of sorbate with a glassy polymeric material below the glass transition temperature (Tg). Originally developed to describe sorption phenomena with glassy polymers (Vrentas and Vrentas 1996), it has subsequently been applied to humic soils (Lu and Pignatello 2002) and more recently to plant fibres and wood (Hill et al. 2009). Briefly, the model considers the creation and destruction of molecular scale nano-pores within the matrix of the material. Below the Tg of the matrix, the opening and closing of these nano-pores is inhibited on the time scale of molecular diffusion within the matrix. Thus, as water molecules diffuse into the matrix, there is a time-lag in the opening up of the nano-pores and as sorbate molecules leave the sorption sites there is a similar lag in the collapse of the nano-pores (Lu and Pignatello 2002). As a consequence the adsorption and desorption processes take place in a material that is effectively in two different states and it is this that gives rise to the phenomenon of irreversible sorption (hysteresis). Certain predictions can be made from this model. A higher degree of hysteresis will be

observed with materials that have higher cross-link densities within the matrix, hence a more lignified material would be expected to show more hysteresis – as is observed with coir (Fig. 1a) and also with thermally modified wood (Fig. 1c). Secondly, as the isotherm temperature moves closer to the Tg of the matrix, a reduction in the size of the hysteresis loop will occur, as is seen in Fig. 1c.

#### Sorption kinetics

It has been shown that the sorption kinetics of water vapour interacting with wood and plant fibres obeys the parallel exponential kinetics model (Hill et al. 2010). This has the form shown in Eqn. 1.

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

Where MC is the moisture content of the sample at time t,  $MC_0$  is the moisture content of the sample at time zero,  $MC_1$  is the moisture content at infinite time associated with the fast kinetic process,  $t_1$  is the characteristic time of the fast process,  $MC_2$  and  $t_2$  are the equivalent parameters for the slow process. Hence a typical adsorption or desorption kinetics curve can be deconvoluted into two curves (one 'fast' process and the other 'slow' process) as shown in Fig. 2a. It is clear that the sorption kinetics process is definitely non-Fickian in nature and that the sorption kinetics can be explained as being due to polymer relaxation processes within the cell wall matrix (Hill and Xie 2011).



**Fig. 2.** A typical sorption kinetics curve deconvoluted into fast and slow exponential processes (a), two Kelvin-Voigt elements in series (b).

The model used involves two Kelvin-Voigt elements in series (Fig. 2b), each element having a kinetic response described by Eqn. 2.

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

Where  $\varepsilon$  is the strain (here assumed to be equivalent to the mass change),  $\sigma_0$  is the applied stress, *E* is the modulus of the matrix and  $\varphi$  is a time constant which is defined as the ratio  $\eta/E$ , where  $\eta$  is the viscosity. The applied stress is taken to be the same as the pressure exerted within the matrix by the presence of sorbed water molecules ( $\sigma_0 = \Pi$ ), which can be calculated from the following expression (Eqn. 3).

$$\Pi = -(\rho/M) \operatorname{RT.ln}(p_i/p_f) \tag{3}$$

Where  $\Pi$  is the swelling pressure,  $\rho$  is the density of water, M is the molar mass of water, R is the universal gas constant, T the absolute temperature,  $p_i$  the initial pressure of the atmospheric water vapour,  $p_f$  the final pressure of the atmospheric water vapour. By inspection of Eqn. 1 and Eqn. 2, it can be seen that the moisture content of the material is determined by the modulus (high modulus means low moisture content) and by the applied swelling pressure (equivalent to the atmospheric relative humidity) (MC =  $\sigma_0/E$ ). The role that OH group concentration has in determining moisture content is not defined. Clearly, the OH groups are responsible for making the material hygroscopic, but the EMC seems to be a function of cell wall modulus only.

When the sorption kinetics fitting parameters are used as input into the K-V model, plots such as those shown in Figure 3 are obtained.



**Fig. 3.** Plots showing variation in matrix modulus (a) and viscosity (b) for Scots pine obtained from sorption kinetics experiments.

The values obtained for the cell wall matrix modulus are reasonable and in agreement with nano-indentation measurements. The dramatic decline in the modulus as RH increases is interpreted as being due to plasticisation of the cell wall matrix macromolecules with sorbed water. The values for viscosity are reasonable for what might be measured for a solid, although viscosity measurements using static relaxation measurements have not yet been performed to confirm the values obtained. There is a greater difference found in the modulus obtained from adsorption measurements compared to adsorption, although this has been found to vary between species and is dependent upon any modification that may have been performed on the material. With viscosity, it is the slow process viscosity that shows a large difference between adsorption and desorption. This may perhaps be related to the hysteresis phenomenon and this is the current direction of our research.

#### CONCLUSIONS

The water sorption behaviour of various plant materials has been studied and it is apparent that the amount of moisture sorbed is a function of the stiffness of the cell wall. It is also observed that cell wall stiffness is a factor in determining the size of the area bounded by the hysteresis loop. The sorption kinetic behaviour has been found to be very accurately described by the parallel exponential kinetics model. This can be further interpreted as being a relaxation limited kinetic behaviour. Thus, a link between sorption kinetics and hysteresis appears likely. Analysis of the sorption kinetics parameters in terms of two series linked Kelvin-Voigt elements yields values for cell wall modulus and viscosity that appear to be reasonable.

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# SORPTION BEHAVIOUR OF SCOTS PINE IN NORTHERN EUROPE

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### ABSTRACT

Wood as a hygroscopic material gains or loses moisture with changes in climate of the surrounding air. The moisture content influences strength properties, hardness, durability and machinability. Therefore the hygroscopicity is a very important property, last but not least for economic factors. Below fibre saturation, a change in moisture content causes shrinkage or swelling and anisotropic behaviour can be seen in the different growth directions. For a better understanding of the sorption behaviour of Scots pine (Pinus sylvestris L.) the variation between different adsorption and desorption curves has been investigated. Trees from 25 different sites in Northern Europe were collected and 3651 samples (1510 heartwood- and 2141 sapwood-samples) measuring 5 (T) x 10 (R) x 30 (L) mm were obtained. The sorption isotherms for all specimens were measured at 25 °C at relative humidities of 15, 35, 55, 75 and 95 % for both desorption and adsorption. The aim of this study is to investigate the influence of raw material variability on the sorption behaviour of Scots pine. Due to the different growing conditions, densities and wooden material (heart or sapwood) variations within the sample groups have been found. Correlations between moisture and density respectively latitude were investigated.

Keywords: Geographic origin, isotherm, moisture sorption, Scots pine

#### INTRODUCTION

Scots pine is adapted to a wide variety of climates and growing conditions as indicated by its extremely large natural range. It grows naturally from Spain to the Pacific Ocean and from above the Arctic Circle in Scandinavia to the Mediterranean. It is the most widely distributed pine in the world and primarily used as pulpwood and saw logs (Burns et al. 1990). Due to the wide natural distribution one can find a large variation within the material. This applies to the chemical composition as well as to the woodwater relation. Wood as a hygroscopic material gains or loses moisture with changes in relative humidity of the surrounding air. The moisture content influences strength properties, hardness, durability and machinability. Therefore knowledge regarding the relationship between water and the wood structure is of great value.

The total sorption of wood is indicated by the sorption capacity of the main components. Hydroxyls are the most important groups involved in the sorption process

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accompanied by carboxylic groups. Due to the structure and available hydroxyl groups, cellulose contributes 47 %, hemicelluloses 37 % and lignin 16 % to the sorption (Kollmann 1968). According to Stamm (1964) water is mostly sorbed in the noncrystalline segments of the cellulose chains and hemicelluloses. The adsorption of vapour molecules is an accumulation to a surface, while sorption mechanisms have physical or chemical nature. The chemically bound water is compressed to a density of 1.3 g/cm<sup>3</sup>.

In a living tree moisture contents of more than 100 % can be reached in the sapwood (Fromm et al. 2001) and the water is found as bound water in the cell walls and free water in the cavities. After the tree is felled wood loses moisture until equilibrium with the surrounding climate is reached (Time 1998). The equilibrium moisture content (EMC) of wood at a given relative humidity (RH) is a function of species, location within the tree, stress, history and temperature (Skaar 1988). Below the fibre saturation point the bound water will begin to evaporate from the cell wall (desorption) while drying and accordingly, an increase of RH causes an uptake of water into the cell walls until the new EMC is reached (adsorption). In the range between oven-dry and the fibre saturation point the relationship between EMC and RH at a constant temperature is called sorption isotherm and it exhibits a sigmoid sorption curve (Rowell 2005). There is a significant difference between the sorption isotherms of de- and adsorption where the desorption curve runs above the adsorption curve (Skaar 1988). This phenomenon is called hysteresis and is the result of the linkage of free hydroxyls of the wood when there is very little moisture in wood. Hence, during the following adsorption the number of available hydroxyls is generally smaller (Tsoumis 1991).

The variations of the EMC at a given RH are caused by the different species, heart- and sapwood, the composition and proportions of cell-wall constituents and extractives (Skaar 1988). In general, the ability to transport water decreases with increased density and extractive content. Regarding Scots pine, its heartwood is known to have an extractive content twice as high as the sapwood (Sehlstedt-Persson 2001).

While Morén and Sehlstedt (1984) found no correlation between moisture content and density a significant difference in EMC for heart- and sapwood was discussed by Koponen (1985). An answer to the question if a relation can be found if one considers the available data of different sites could help to understand and classify the influencing factors on the wood-water relation for Scots pine.

## MATERIAL AND METHODS

#### Wood material collection

The sample material used in this study was collected during autumn and winter 2009/2010 (Behr et al. 2010, Zimmer et al. 2010). Scots pine samples from 25 different sites in Estonia, Finland, Lithuania, Norway, Scotland and Sweden were collected and used to study the variation within the material. Different properties like latitude, longitude, altitude, diameter, tree height, age and site index (Norwegian site index for Scots pine: H40 = height of tree at age 40) were noted. All in all 225 logs have been collected, heart- and sapwood were separated and radial layers were produced. From these layers clear mini block samples (seven per layer) were cut. The samples were free from wood defects and had a perpendicular annual ring orientation. 3651 samples (1510 heartwood and 2141 sapwood samples) were obtained.

#### **Sorption measurements**

After cutting the samples were dried at 50 °C for two days, to assure all the samples to be on an adsorption curve during the first sorption step (15 % RH). The samples were stored in different climates at a temperature of 25 °C and relative humidities of 15, 35, 55, 75 and 95 %, until the respective EMC were reached according to EN 13183-1: 2002. For calculating the EMC the samples were dried at 103 °C in a drying-oven until constant weight was reached (Eq. 1).

$$EMC = \frac{W_u - W_0}{W_0} \times 100 [\%]$$

$$W_u \quad \text{wet weight [g]}$$

$$W_0 \quad \text{dry-weight [g]}$$

$$Eq. 1$$

According to EN 13183-1: 2002, constant weight was considered to be reached when the mass change of the samples within 6 h had been less than 0.1 % (in relation to the previous measurement). The climate in the chamber was constantly controlled with an ElproEcolog TH2. The conditioning was performed with a climate chamber (Termaks KBP 6395 F). For weighing a Mettler Toledo XP 205 Delta Range (0.15 mg resolution) was used.

#### **Data Analysis**

The data collection and the first preparation were performed with Microsoft's Excel 2003. For the statistical analysis the JMP Pro 9.0.0 (2010) software from SAS Institute Inc. (Cary, North Carolina) was used. The Mahalanobis and Jackknife distance outlier analysis was performed before the analytic statistical analysis.

#### **RESULTS AND DISCUSSION**

A variation in sorption behaviour within the material was found. In average sapwood shows a higher moisture content than heartwood (Table 1).

		Hear	twood	Sap	wood	Difference of EMC	
	RH [%]	Mean EMC [%]	Standard Deviation	Mean EMC [%]	Standard Deviation	heart- to sapwood [%]	
u	15	4,27	0,38	4,70	0,26	9,08	
tio	35	6,72	0,33	7,25	0,24	7,28	
orp	55	9,41	0,32	10,08	0,26	6,57	
Ads	75	13,29	0,42	14,09	0,34	5,68	
V	95	22,84	1,15	23,83	0,79	4,18	
ion	75	16,00	0,49	16,66	0,34	3,98	
rpti	55	11,24	0,32	12,25	0,26	8,31	
esol	35	8,24	0,30	8,65	0,25	4,82	
Á	15	5,61	0,45	5,44	0,25	3,09	

Table 1. EMC for de- and adsorption; heartwood and sapwood.

Additionally, a variation within a sample group of heart- and sapwood for each sorption step was found. Figure 1 shows exemplary the variation of the moisture content for heartwood at 55 % RH adsorption with a mean value and a median of 9.4 %.



Fig. 1. Moisture content distribution for heartwood at 55 % RH adsorption.

While the bivariate analysis of moisture content [%] by density shows no correlation ( $R^2 < 0.1$  for heart- and sapwood at each sorption step), the correlation of water content [g] to the density is significant (Figure 2 and 3). Heartwood shows at each sorption step a lower regression coefficient  $R^2$ . An increase of density causes a larger quantity of available functional groups. Especially hydroxyls (mainly holocellulose) are responsible for the attraction of water molecules by the wooden structure (Tsoumis 1991). The ratio of hydroxyls to attracted water molecules at a constant relative humidity is not influenced by the density, it remains constant. Due to this fact the absolute amount of water [g] has to be used for a correlation with density.



Fig. 2. Bivariate analysis of water content by density at 55 % RH adsorption (heartwood).

water content [%] = 0.0027177 + 0.1340351 \* ovendry density [g/cm<sup>3</sup>]  $R^2 = 0.87$ 



Fig. 3. Bivariate analysis of water content by density at 55 % RH adsorption (sapwood).

water content [%] = 0.00921 + 0.1255681 \* oven-dry density [g/cm<sup>3</sup>] R<sup>2</sup> = 0.90

The large sample area included different latitudes (from 55°44N in Lithuania to 66°52N in Norway). A oneway analysis of variance of water content by latitude groups shows a weak trend (Figure 4). The lower water content in higher latitudes can be seen in all sorption steps for both, heart- and sapwood.



**Fig. 4.** Oneway analysis of variance of water content by latitude at 55 % RH adsorption (heartwood) and comparison of all pairs using Tukey-Kramer HSD ( $R^2 = 0,11$ ). Each group represents stands sorted by latitude. The latitude increases from group a to group d.

Comparing the oven-dry density of the heartwood samples in relation to the latitude shows that northern stands seem to have a lower density (Figure 5). This verifies the lower water content in the same group.

The higher extractive content in the heartwood seems to lead to lower moisture contents. The specific extractive content of the samples has not been investigated yet. An increase in extractive content however causes a reduced water uptake (Sehlstedt-Persson, 2001). Due to the lower  $R^2$  for the heartwood samples extractives seem to mask the density which has been also discussed by Posey et al. (1970). To investigate the influence of the density the absolute water content has to be considered. The low water content in the northern stands verifies the low densities in these groups. Besides the latitude the relation of water content with altitude, longitude and site index was investigated. While altitude and longitude have no influence on the water content the site index shows a weak trend: a large site index seems to lead to a higher water content.



**Fig. 5.** Oneway analysis of oven-dry density by latitude (heartwood;  $R^2 = 0.13$ ).

### CONCLUSIONS

The sorption behaviour of Scots pine shows large variations between heart- and sapwood as well as within a sample group. The extractive content seems to have a
strong influence. For a better understanding of the wood-water relation further investigations, especially in regards to the specific extractive content are necessary.

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# DETERMINATION OF WOOD MOISTURE PROPERTIES USING CT- SCANNER IN A CONTROLLED ENVIRONMENT

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## ABSTRACT

The aim of the present work was to examine the existing algorithm for the moisture content calculation and also to use this algorithm to analyze and compare the moisture flow data for high and low temperature drying. The use of the existing algorithm for the dry weight moisture content on density data from the CT-scanning during high and low temperature drying in the climate chamber showed that this method is a powerful tool for analyzing the moisture flow inside the wood piece. Furthermore, the new CT-scanner together with the climate chamber gave unique results, as it has not been possible to study high temperature drying with this method before.

Key words: CT- scanning, high temperature drying, low temperature drying, image processing.

### INTRODUCTION

In 1992 an X-ray computed tomography (CT)-scanner was installed at Luleå University of Technology in Skellefteå. It has been used since then for advanced non-destructive studies of different kinds of wood internal characteristics. By using the CT-scanner, Lindgren (1992) showed a correlation between CT-numbers, wood density and dry weight moisture content (mc) values. Furthermore, Lindgren (1992) also showed the accuracy between real values and CT measurements. Also, other researchers at the Division of Wood Physics, Luleå University of Technology in Skellefteå, have been using the CT-scanner in their research. Wiberg (2001) analyzed moisture distribution and moisture flux above the fibre saturation point (fsp) for different kinds of wood during drying. All the measurements were done in a simply designed climate-controlled tube, where the maximum temperature that could be reached was 70°C. CT-scanning during low temperature (LT) drying of birch and *Pinus radiate* pieces was described by Scheepers (2006). The data was collected to determine mc, moisture loss from the core of the wood pieces. CT-scanning during LT-drying has been done by Sehlstedt- Persson

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(2008) as well, by weighing the wood mass and measuring the temperature of wood during LT drying for calculating the position of the evaporation front beneath the surface. These measurements were made for comparisons with CT-scanned density pictures of dry shell formation. To determine physical parameters for wood the wet and dry wood densities need to be known when developing physical computational models. By using the CT-scanner, Lundgren (2007) and Hansson (2007) measured the internal structure of density in pieces of wood for input to finite element models. In 2008 the old CT-scanner at Luleå University of Technology in Skellefteå was replaced and at the end of the year 2010-2011 a new purpose-built climate chamber was installed as a complement to the CT-scanner.

The aim of the present work was to examine the existing algorithm for the moisture content calculation. To analyze and compare the moisture flow data for high and low temperature drying.

## MATERIAL AND METHODS

### **Principles of a CT-scanner**

A CT-scanner simply works by passing X-rays through the wood and receiving information with a detector on the other side. The X-ray source and the detector are interconnected and rotate around the wood specimen during the scanning period. X-rays are electromagnetic waves. A digital computer collects the data that is obtained and then integrates it to provide a cross-sectional image (tomogram) that is displayed on a computer screen. High density wood or wet wood appears white on a CT image, while the dried wood appears darker grey.

### Principles of climate chamber and the control schedule

The climate chamber is designed in order to work with the CT-scanner. In simple terms, the climate chamber is designed with an inner and outer tube, where the air, driven by a fan, flows through the heating coils and then into the inner tube, where the material is placed for the drying. In the end of the inner tube, the air flow turns back to the fan by flowing in the outer tube. For increasing the humidity in the chamber a steam generator is used. The climate chamber could also make conditioning by water spray and steam in order to equalize the moisture gradient, which may have been caused by intense drying. High and low temperature drying processes could be done in the kiln. The control schedule, which has been developed by Valutec AB (2010), is built up by assigning different control parameters in different phases.

### Algorithm for moisture content calculation

Since wood starts to shrink below the fsp during drying, the geometrical shape of the wood piece will change. This means that more thorough transforming must be done on the dry wood image to the shape of the wet wood image prior to calculating the mc (Hansson 2007). By using bidirectional elastic registration (Arganda-Carreras et al. 2006), the dry wood image can be transformed to the shape of the wet wood image. This algorithm (bUnwarpJ) for elastic and consistent image registration is developed as a plug-in to the software ImageJ (Image processing and analysis in Java). It is performed

as a simultaneous registration of two images, in this case the dry and wet wood images. The dry wood image is elastically deformed in order to look as similar as possible to the wet wood image. Two images are produced as a result: the deformed versions of dry and wet wood images. By using the same approach as Hansson (2007), the transformation of the CT-images and the moisture content calculation was done. From the algorithm, mc distribution was calculated for test pieces.

### High temperature and low temperature drying

For the high temperature experiment, sapwood pieces with dimensions of 35x70x400 mm and 24x66x400 mm were freshly cut out from logs of pine. For low temperature drying, two sapwood pieces with the dimensions 26x92x400 mm and 26x98x400 mm were cut out from the board that was brought from the Martinssons sawmill. The ends were sealed with one layer of silicon closest to the wood surface and then one layer of aluminium foil followed by an additional layer of silicon. During the drying process, the temperature of the wood pieces was measured in two positions by thermocouples, in the core of the board and 5 mm below the surface. The thermocouples were installed into drilled holes and sealed with silicon. For the high temperature experiment, the pieces were dried at 90/110°C (wet bulb/ dry bulb temperature), and for low temperature drying 56/80°C (wet bulb/ dry bulb temperature) for 50 hours. To reach the dry density, the pieces were dried in 103°C for 24 hours. The boards were CT-scanned, 10 cm of the middle part with a 5 mm slice width every 15 minutes. Thus 20 density images were obtained for each time period. With the previously described algorithm, the mc distribution was calculated for each time step. Furthermore, the average mc and density for a selected core region were determined for every time step. The selected core region had a dimension of 130x20 pixels (75.4x11.6 mm) for HT and 120x20 (69.6x11.6 mm) pixels for LT. Hence, the rate of the moisture flow from the core vs. the average mc could be calculated. The moisture flow is calculated by taking the difference between the densities for each time step.

### **RESULTS AND DISCUSSION**

### High temperature drying

By using the method for calculating the dry weight moisture content, the moisture loss from the core could be calculated (Fig. 1).



**Fig. 1.** The rate of moisture loss from the core vs. the average dry weight moisture content (left) and the core temperature vs. time (right).

The rise in the temperature in the kiln makes a big difference between the wood core and the dry bulb temperature inside the kiln (Fig. 1). As the temperature increases, therefore the density decreases and this creates a pressure difference. Since the temperature is proportional to pressure, the water and vapour pressure is much higher in the region close to the surface than to the wood core. The temperature difference makes the moisture flow to the internal part of the wood piece. This phenomenon is called Darcy flow, which explains the negative moisture flow at the beginning of the drying (Fig. 1) as well as the increasing density in Fig. 2. After about half an hour, the wet bulb temperature in the kiln becomes equal to the wood temperature. Furthermore, the water starts to strive towards the surface from the core, as it begins to evaporate from the surface.



Fig. 2. Density vs. time and dry moisture content profiles.

The rising core temperature after around half an hour led to an accelerated moisture loss from the core. The evaporation of water from the surface sets up capillary forces that exert a pull on the free water in the zones of wood beneath the surfaces. When there is no longer any free water in the wood, capillary forces are no longer of importance. The drying process is now in the capillary phase and this phase ended approximately after 4.5 hours. By studying the density change (Fig. 2), one can see that it decreases linearly in the defined core area from about 0.5 hours to 4.5 hours, which means that the moisture inside the wood piece decrease almost uniformly as long as there is capillary communication.

The average moisture flow vs. dry moisture content (Fig. 1) shows that the moisture flow constantly decreases from 0.8 to around 0.3. The core temperature starts to rise after 5 hours, which means that the drying process is going into the transition phase and the rate of moisture loss from the core vs. the average dry weight moisture content will drop dramatically (Fig. 1). Furthermore, the moisture flow starts to decrease. Hence, the capillary connection has begun to break down. After the transition phase, with about a 0.28 dry weight moisture content, the core temperature increases faster (Fig. 1) and the rate of the moisture loss from the core vs. the average dry weight moisture (Fig. 1) and the rate of the moisture loss from the core vs. the average dry weight moisture (Fig. 1) is almost constant. Furthermore, the density vs. time at present only slowly decreases. The drying process is now in the diffusion phase and moisture slowly evaporates from the wood piece.

### Low Temperature drying

By using the method for calculating the dry weight moisture content, the moisture loss from the core can be calculated (Fig. 3).



**Fig. 3.** The rate of moisture loss from the core vs. the average dry weight moisture content (left) and the core temperature vs. time (right).

When comparing the rate of moisture loss from the core vs. the average dry weight moisture content for high temperature and low temperature drying, the low temperature drying has only a small negative moisture flow at the beginning of the drying process (Fig. 3). The shape of moisture flow from the core vs. the average dry weight moisture content plot for the LT drying process looks similar to the HT drying process. The density change level is a little bit higher in the HT plots (Fig. 4). The rising core temperature after about half an hour in the LT drying process led to accelerated moisture loss from the core, as in the HT drying process. The drying process is now in the capillary phase and this phase ended after approximately 7.5 hours. By studying the density change (Fig. 4), one can see that it decreases linearly in the defined core area from about 0.5 hours to 7.5 hours, which means that the moisture inside the wood piece decrease almost uniformly as long as there is capillary communication. The drying process was disturbed at the beginning of the test, since there was a problem with the CT-scanner. The drying chamber was shut down for some time to fix the scanner. That is the reason for the disturbance that appears in the temperature vs. time plot (Fig. 3) at the beginning of the drying process.



Fig. 4. Density vs. time and dry moisture content profiles.

The average moisture flow vs. dry moisture content (Fig. 3) shows that the moisture flow constantly decreases from 0.6 to around 0.3. The core temperature starts rising after 8 hours, which means that the drying process is going into the transition phase and the rate of moisture loss from the core vs. the average dry weight moisture content drops dramatically (Fig. 3). Furthermore, the moisture flow starts to decrease.

After the transition phase, with about a 0.28 dry weight moisture content, the core temperature increases faster (Fig. 3), and the rate of the moisture loss from the core vs. the average dry weight moisture (Fig. 3) is almost constant. Furthermore, the density

change vs. time only slowly decreases. The drying process is now in the diffusion phase and moisture slowly evaporates from the wood piece.

### CONCLUSIONS

The use of the existing algorithm for the dry weight moisture content on density data from the CT scanning during high and low temperature drying in the climate chamber showed that this method is a powerful tool for analyzing the moisture flow within the wood piece. The new CT-scanner together with the climate chamber gave unique results, as it has not been possible to study high temperature drying with this tool before.

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# WATER ABSORPTION IN COATED WOOD - INFLUENCE OF DIFFERENT WOOD TYPES AND COATINGS

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### ABSTRACT

Coated specimens of fast-grown and slow-grown Norway spruce (*Picea abies* (L.) Karst) and of Scots pine heartwood (*Pinus Sylvestris* L.) were subjected to liquid water exposure based on the European standard EN 927-5. The exposure time was prolonged and both amount of absorbed water and moisture content gain was studied after different time intervals up to 18 weeks. Waterborne top coat without primer led to more water absorption than waterborne top coat with solvent-borne primer, and solvent-borne top coat with no primer led to the smallest amount of absorbed water. The performances of the coatings relative to each other were similar within each wood type. On average the coated Scots pine heartwood had lower uptake of liquid water and lower moisture content gain as well as amount of absorbed water was of value in understanding the differences between wood types.

Key words: Liquid water absorption, coated wood, wood properties, Norway spruce, Scots pine, heartwood

### INTRODUCTION

Coating of wood in exterior claddings is done mainly to obtain the desired aesthetical appearance and to protect the wood from water uptake and other external stress (de Meijer 2001; Sell 1975). Water is a key factor, both regarding microbial deterioration and dimensional stability of wood. The liquid water permeability of different coatings on a single type of wood substrate has been extensively studied (e.g. Ahola et al. 1999; de Meijer et al. 2001; de Meijer and Militz 2000; Derbyshire and Miller 1997; Ekstedt 2003). Some studies have included comparison of wood species and/or effects of preweathering (Ahola 1991; Ahola et al. 1999; de Meijer and Militz 2001; Williams and Feist 1994). Liquid water permeability measured according to the standard EN 927-5 has proven to correlate well with moisture content in full scale window frames in service (de Meijer 2002). Knowledge on the performance of wood qualities in interaction with different coatings during long-term exposure to liquid water is limited.

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The objective of the present work was to study the effect upon long-term water absorption by different wood types in combination with different coatings, and to investigate the effect of reporting results in terms of either mass of water absorbed per area or moisture content gain in the wood specimens.

## MATERIAL AND METHODS

The material was Norway spruce and Scots pine (*Pinus sylvestris* L.) sampled from air dried centre boards, sawn from butt logs in mature trees. The spruce material was sampled from slow grown, high density (SH) and fast grown, low density (SL) Norway spruce. The SH specimens were collected from two Norway spruce stands in northern Norway. The SL specimens were collected from a single stand in south-eastern Norway. The Scots pine heartwood (PH specimens) was collected from four mature pine stands in south-western Norway. All stands were located at altitudes below 300 m.

Preparation of wood specimens is described in Sivertsen and Flæte (2011). Annual ring width and density (at 12 % moisture content) of the test specimens are given in Table 1.

**Table 1**: Annual ring width (ARW) and density at 12 % MC (D12) of the specimens in the sample groups. N = number of specimens in each group. SL = spruce with wide annual rings; SH = spruce with narrow annual rings; PH = pine heartwood. SB = solventborne coating; WB = waterborne coating; PW = waterborne coating with solventborne primer; U = uncoated.

Wood type	Conting	Ν	$D12 (g/cm^3)$		ARW (mm)	
wood type	Coating		Mean	St.dev	Mean	St.dev
SL	SB	6	0.364	0.007	4.4	0.9
	WB	6	0.361	0.011	5.0	0.7
	PW	6	0.369	0.012	4.5	0.8
SH	SB	6	0.522	0.031	1.3	0.4
	WB	6	0.518	0.032	1.3	0.4
	PW	6	0.522	0.031	1.3	0.3
PH	SB	6	0.480	0.080	2.0	0.6
	WB	6	0.463	0.045	1.9	0.7
	PW	6	0.485	0.078	1.7	0.7

The test specimens were re-conditioned to 20°C and 65 % RH before sealing with two layers of a two component solvent-borne lacquer (Pyrotect 2K (Rütgers Organics)) on ends, edges and back face. Two coatings with different permeability properties were applied; a waterborne top coat (WB) and a solvent-borne top coat (SB). In addition, a system with a solvent-borne primer and the same waterborne top coat (PW) was applied, in order to investigate the impact of the primer on liquid water permeability. All the coating products were commercially available. The coatings were applied in two layers and the primer in one layer according to the instructions given by the manufacturer. In order to leach any water-soluble components the specimens were subjected to three cycles of 72 hours wetting and 96 hours drying prior to the final test, and re-conditioned at 20°C and 65% RH to stable mass.

The specimens were placed test face down in de-ionised water as described in the standard EN 927-5. The coated specimens were exposed to water for 3024 hours (18 weeks. Data after 72 hours (3 days) and 672 hours (4 weeks) of absorption were used in analyses of absorption performance.

The water absorbed by each specimen was measured as mass of absorbed water per test face area (MWA,  $g/m^2$ ) relative to the weight of the conditioned specimen prior to the test, in accordance with EN 927-5 (Eq. 1).

$$MWA_1 = \frac{(w_1 - w_0)}{A} \tag{1}$$

where  $MWA_1 = mass$  of absorbed water per area (g/m<sup>2</sup>) at time 1;  $w_0 = weight$  (g) at time 0;  $w_1 = weight$  (g) at time 1; A = area of test face (m<sup>2</sup>).

The moisture content gain in each specimen (MCg, %) was calculated according to Eq. 2. The MCg was chosen in order to be able to evaluate the changes in moisture content as a result of the absorbed water.

$$MCg_1 = \frac{w_1 - w_0}{w_d} \times 100$$
 (2)

where  $MCg_1$  = moisture content increase at time 1(%);  $w_0$  = weight (g) at time 0;  $w_1$  = weight (g) at time 1;  $w_d$  = dry weight, untreated (g, calculated based on the MC of the reference specimen).

The statistical analyses were performed using JMP software (SAS Institute Inc 2010).

### RESULTS

Figure 1 shows the development in MWA for the different coatings within each wood type. WB specimens had highest MWA within the spruce wood types (Figure 1 a, b). Coating type appeared to have little effect on MWA in pine heartwood specimens, but SB was slightly higher than WB, and PW slightly lower (Figure 1 c).



**Fig. 1**. Development in weight gain  $(g/m^2)$  during the entire experiment shown as mean curves for each combination of wood type and coating. **a**: SH specimens, **b**: SL specimens, **c**: PH specimens.

PH specimens had lower water uptake than all spruce specimens after 72 hours (Table 2). After 672 hours, SH specimens had higher MWA than SL or PH specimens regardless of coating type, and the difference was significant for PH/SB and PH/PW specimens. As can be seen from the right half of Table 2, SL specimens clearly had higher MCg than other wood types both after 72 and 672 hours.

Density had no significant effect on MWA after 72 or 672 hours, but after 1008 hours density (p < 0.0001, F-ratio 33.3) had larger effect on MWA than coating (p = 0.0033, F-ratio 6.5) or wood type (p = 0.0018, F-ratio 7.3). Regarding MCg density was the most important effect after both 72 and 672 hours, followed by wood type. In Tukey HSD tests on wood types the difference between SL and SH specimens was not significant when the density effect was accounted for, but PH specimens had significantly lower MCg than other wood types both after 72 and 672 hours.

**Table 2.** Results from Tukey HSD analyses (Ty) on combinations (Comb.) between coating types and wood types. MWA and MCg after 72 and 672 hours. SL = spruce with wide annual rings; SH = spruce with narrow annual rings; PH = pine heartwood. SB = solventborne coating; WB = waterborne coating; PW = waterborne coating with solventborne primer.

	72 h			672 h	L		72 h			672 h	
Comb.	Ту	LSMean	Comb.	Ту	LSMean	Comb.	Ту	LSMean	Comb.	Ту	LSMean
MWA (g	$(/m^2)$					MCg (%	)				
WB,SH	А	105	WB,SH	А	630	WB,SL	А	1.44	WB,SL	А	7.85
PW,SH	AB	93	PW,SH	В	546	PW,SL	А	1.40	PW,SL	А	7.54
WB,SL	AB	92	SB,SH	BC	533	SB,SL	AB	1.33	SB ,SL	А	7.29
PW,SL	В	90	WB,SL	BC	505	WB,SH	BC	1.14	WB ,SH	AB	6.85
SB,SL	BC	88	WB,PH	BC	496	WB,PH	С	1.02	WB,PH	BC	5.98
SB,SH	BC	88	PW,SL	BC	489	PW,SH	С	0.98	PW,SH	BC	5.75
WB,PH	BC	85	SB,SL	BC	483	PW,PH	С	0.95	SB,SH	BC	5.74
PW,PH	BC	82	PW,PH	С	479	SB,SH	С	0.94	SB,PH	С	5.70
SB,PH	С	74	SB,PH	С	473	SB,PH	С	0.90	PW,PH	С	5.52

The sequence of coating types regarding MWA was the same within all wood types (Table 2). Specimens with WB coating absorbed more water than specimens with PW coating, while specimens with SB coating absorbed the smallest amounts of water. In a separate Tukey HSD test on coatings after 72 hours, the difference between WB and SB coated specimens was significant, while PW specimens were not significantly different from the others. After 672 hours WB specimens were significantly different from SB and PW samples regarding MWA, but SB and PW specimens were not different from each other. Similar results were found for MCg as for MWA; WB coated specimens gained significantly more in MC than SB coated specimens, while PW coated specimens were not different from the other coatings.

### DISCUSSION

The results from this study are in line with earlier studies (e.g. Greystone and Ekstedt 2004) showing higher water permeability in waterborne acrylic coatings than in solventborne coatings. The fibre saturation point (FSP) has been suggested as the upper moisture content limit for moisture penetrating through a surface coating (Derbyshire and Miller 1997). The explanation for this has been proposed to lie in the transition from moisture movement by diffusion to the very low rates of cell-wall flow above FSP (Derbyshire and Robson 1999). Our results seem to confirm this notion.

For Norway spruce the different MWA found for coated SH and SL specimens has earlier been shown to be a density effect (Sivertsen and Flæte 2011), due to the lower amount of water needed to reach fibre saturation in specimens with lower density. As the wood material approaches FSP the absorption into the wood will decrease to a point where it is smaller than the transmission rate through the coating, and the water flux will decrease. When the entire piece of wood has reached FSP the moisture concentration will be 100 % of the potential both on the inside and the outside of the coating and the water flux will stop, provided that a diffusion tight sealant prevents moisture escape through the sides and/or back face. Thus, the total amount of water absorbed in wood with a coating that prevents capillary transport can be expected to be higher in wood with higher density (Greystone 2001). The absorbed water caused SL specimens to increase more in moisture content and reach values close to fibre saturation point earlier than the SH specimens.

On average PH specimens had the lowest MWA of all the wood types after 72 and 672 hours. As can be seen from Figure 1 (b, c) PH specimens had slightly higher MWA at the end of the experiment than SL specimens, but the difference was not significant. The mean density in PH specimens was substantially more different from SL than from SH specimens (Table 1). Based on the effect of density on water flux found for Norway spruce a larger difference between PH and SL than between PH and SH specimens could be expected. As significant different water uptake was found only between PH and SH specimens, density cannot explain the difference. The FSP in pine heartwood is lower than that in pine sapwood or spruce (Kollmann and Côté 1968), and as the water flux depends on the moisture concentration gradient, lower FSP should cause lower water flux. In addition, low diffusivity in the wood will reduce the water flux. The diffusivity in heartwood of Scots pine has been shown to be lower than in sapwood (Sehlstedt-Persson 2001), due to the high extractive content.

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# WOOD WEATHERING FROM A SERVICE LIFE PERSPECTIVE

## Rüther, P.<sup>1</sup>

## ABSTRACT

Untreated wooden cladding has a long tradition and has in recent years become a both economically and environmentally beneficial solution in miscellaneous modern building applications. Untreated wood in cladding and similar applications represents a building part that changes its appearance rather dramatically without compromising its technical functionality. The aesthetic service life is often the decisive criterion for these applications. This paper presents a study on the weathering of untreated, i.e. unpainted wood. Following the service life prediction methodology suggested in the ISO 15686 standard, wood was weathered both outdoors and in two laboratory weathering apparatuses. Climate data for the test site were assessed including temperature, winddriven rain and solar radiation. The performance of non-structural wood components in exterior above-ground applications is often closely related to the aesthetics of the wooden component in question. Hence, a method for color determination of large samples was developed, and the topic of human color perception is discussed briefly. It was found that the colonization by mould growth fungi contributes significantly to its surface appearance. Differences between materials and exposure directions were investigated. The topic of limit-state for aesthetic service life is discussed and a possible assessment method for such applications is presented. No simple dose-response relationship between solar radiation and wind-driven rain, and color response of the material could be established. Acceleration factors for the conducted laboratory weathering tests are discussed. Furthermore, color changes by outdoor versus laboratory weathering were evaluated. It was found that the conducted laboratory weathering cycles could not recreate the visual appearance of an outdoor weathered surface. In summary, the suggested bottom-up approach of the service life prediction methodology is not easily adaptive for wood in this application.

Key words: Natural weathering, laboratory weathering, color changes, mold growth, aesthetic service life.

### INTRODUCTION

Wood is a versatile material that is widely used both in indoor and outdoor applications, e.g. cladding, deking, and railing. Wood undergoes several transformations during weather exposure. Weathering of wood involves color changes, loss of surface fibres, roughening and cracking of the surface, and changes in chemical composition (Borgin

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1971, Hon and Minemura 2001). Environmental agents, e.g. solar radiation and precipitation have a major role in wood weathering. Color changes are rapidly superposed by mould fungus colonization. (Sell and Leukenes 1969, Wälchli 1969, Kühne et al. 1970). However, durability and serviceability are not usually impaired by weathering. Hence, the service life of a wooden component is often related to its aesthetic appearance, e.g. its color. Weathering studies might be conducted under natural, i.e. outdoor conditions, or artificial, e.g. laboratory conditions. Laboratory weathering is chosen to accelerate or compress degradation processes. However, the spectral distribution of the light source, the composition of the weathering cycles, and the chosen temperature levels have a great influence on the chemical reactions (Arnold et al. 1991). Changes in appearance caused by wood weathering have so far been evaluated subjectively (Sell and Leukens 1971), i.e. by visual inspection. However, color measurement provides the possibility to objectively assess changes in appearance, since lighting variations and differences in the observers' perception are excluded. This is done by describing color development in an objective way by means of the CIE L\* a\* b\* model (CIE 2004). Differences and similarities between outdoor and laboratory weathering are being investigated. Mold growth for different outdoor exposure directions is discussed as well as limit state for aesthetic service life.

### EXPERIMENTAL

Nine different wood species and wood based products are included in this study, five untreated wood species and four materials that are treated by means of different impregnation techniques: Norway spruce (regular and rough surface), Scots Pine heartwood, Aspen, Larch, Kebony Furu, Kebony SYP (Southern Yellow Pine), Royal impregnation (Scots Pine), and Pressure treated Scots Pine. Test specimens used in this study are approximately 150 mm in width. Different lengths are used for different exposures, depending on the available space, 500 mm length for outdoors and in the Rotating Climate Chamber (RCC), and 200 mm for exposure in the ATLAS solar simulator.

### **Outdoor exposure site**

The specimens are exposed on a test house at Voll in Trondheim, Norway. Samples are exposed vertically facing north, south, east and west for 1322 days. Specimens are mounted with both vertically and horizontally oriented annual rings. Additionally, samples of each material were exposed on a rack at the test site facing south with an angle of  $30^{\circ}$  from the ground. Temperature, precipitation, and solar radiation on a horizontal plane are measured on site.

### **Artificial weathering**

Artificial weathering tests were performed in two apparatuses: An *ATLAS* solar simulation chamber and a rotating climate chamber (RCC). The *ATLAS* solar simulator can be programmed according to the users' requirements, while the RCC runs in a fixed cycle that is defined in NT Build 495 (Nordtest 2000). The apparatus consists of a rotating central unit containing the test specimens located on four sides. The central unit rotates 90° every hour, which thus passes the test specimens into four different exposure environments: UV and IR radiation, water spray, frost, and laboratory climate. Four different test cycles were run in the ATLAS solar simulator. Run 1 with 20 hours of

radiation followed by 4 hour of water spray at a temperature during radiation of  $63^{\circ}$ C; for run 2 the same total amount of water spray and radiation was split into 4 blocks at 5 hours of radiation followed by 1 hour of water spray. Run 3 and 4 were based on run 1, only that for run 3 the temperature during radiation was lowered to  $22^{\circ}$ C and for run 4 radiation was reduced by 50% by setting in an UV filter.

### **Color analysis**

The CIE L\*a\*b\* model (CIE, 2004) describes color in terms of its lightness component L\* and two chromatic components, a\* and b\*. According to BS 6923 (1988) color differences can be calculated by determining the Euclidean distance between two colors as

$$\Delta E = ((\Delta L^{*})^{2} + (\Delta a^{*})^{2} + (\Delta b^{*})^{2})^{1/2}$$
 Eq. 1

To determine the color development of the outdoor exposed samples, test specimens are demounted periodically. After conditioning in a standard climate (20°C, 60 % RH) until equilibrium moisture content, samples are scanned. Color values are obtained using *Matlab*<sup>®</sup> corrected by means of a color profile obtained by a scanner target according to ISO 12641 (1997).

## RESULTS

Results consist of color values for all outdoor and laboratory exposed samples and are presented in Rüther (2011), some main findings are discussed below.

### **Outdoor weathering**

Fig. 1 shows the scanned and color corrected images for Scots Pine heartwood, in Fig. 2 (a-c) color values (L\*, a\*, b\*) are plotted against exposure time for Scots Pine heartwood. At the end of the investigated exposure time, only minor difference in lightness values for east, south, and north exposed samples can be observed.



Fig. 5. Scots Pine heartwood, scanned images from th outdoor weathering experiment. Numbers on top indicate exposure time in days.



Fig. 6. Scots Pine heartwood, color values for outdoor an RCC laboratory exposure.

### Mold growth

One major difference between outdoor and laboratory exposure are biological components that result in an additional coloring of untreated wooden surfaces. It was observed that generally more fungal growth appears on samples mounted with vertically oriented annual rings than on samples mounted with horizontally oriented annual rings, se Fig. 7. This observation was confirmed by darker color for samples with horizontally mounted annual ring throughout all wood species investigated. One possible reason is that horizontally oriented annual rings offer an increased surface area with trapped moisture where biological material is more likely to deposit.



**Fig. 7.** Norway Spruce, annual rings horizontally (a) and vertically (b) oriented, east exposure 1322 days.

### Limit state

In many applications, e.g. above-ground the service life of untreated wood cannot simply be quantified by functional parameters. A limit-state for aesthetic appearance might involve a limit value for  $\Delta E$ , the difference in color value, which is constituted by the user. A possible application are color differences within a façade of untreated wooden cladding, as shown in Fig. 8.



**Fig. 8** Different color of Aspen cladding, Tyholt crematory, Trondheim (left) . Larch samples exposed to west, different stages of exposure and respective color values (right).

### MAIN FINDINGS

Color variations within samples exposed to the same strains proved to be minor, despite the fact that no clear wood samples were used. Annual ring orientation (vertical or horizontal) has a significant influence on the color changes after three years of exposure. Microscopy confirmed that on samples with horizontally mounted annual rings, there was a higher density of mold growth to be found. A higher density of mold growth appearing as grey dots on the surface results mainly in a darker color, a lower value for Lightness. There are some differences depending on the exposure direction of the sample: as expected most mold growth was observed on samples exposed facing west and on the rack, least on samples exposed facing south. Several accelerated weathering tests were performed. None could reproduce the surface appearance of the samples exposed to natural weathering. Though, the rotating climate chamber (RCC) seems to be the one that strains the samples in a similar way. Based on the results of the present study, the weathering cycle that gives results closest to those from outdoor weathering was the one with the lowest radiation level and lowest temperatures.

Service life considerations are often based on ascertained boundary conditions, end of service life or a certain material property that has to fulfill certain conditions. For many building materials and parts, the aesthetic condition is the determining function. Hence, a quantitative determination of limit-state values for untreated wood surfaces in terms of the aesthetic appearance is difficult. Absolute color changes of an untreated wooden cladding are not decisive, specially since the *natural* appearance is intended. However, relative color differences within one particular part or building component are most likely not intended. An individual definition of a limit state value for color variations is a possible approach to make subjective performance requirements accessible to objective assessment.

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# OPTIMUM DESIGN GUIDELINES FOR PLYWOOD SANDWICH PANELS

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## ABSTRACT

The current research deals with optimisation of the plywood sandwich panels with corrugated core under four point loading conditions. The finite element model has been extensively verified by strain and deflection measurements obtained from physical tests from sandwich panel prototypes. An overall good correlation between experimental and numerical models has been achieved. The metamodelling methodology utilising the design of computer experiments with mean square error space filling criteria and approximation techniques have been used for optimisation task. Approximation functions have been created to predict response values for combinations of variables not included in initial experimental design. A four design variables have been considered, including geometrical dimensions and the veneers lay-up configurations. As response functions the panel strain, deflection and volume responses has been considered.

Using metamodelling functions optimal cross section parameters have been elaborated for evaluation of most efficient bending stiffness versus weight ratio. For several panels with optimal cross section parameters design guidelines has been elaborated to overview the capabilities of different geometrical configuration for design of allplywood sandwiches panels.

Key words: plywood 3D structure, mechanical testing, four-point bending, metamodelling, optimisation

### INTRODUCTION

As potential direction for plywood industry in new products research and development may be considered lightweight sandwich structures with reduced structural weight and sustaining load bearing capacities close to convened plywood. Such a solution offer development of a material with high specific strength. In particularly improving the strength/density ratio compared to the solid wood. All plywood| sandwich panels consisting of a plywood surfaces and corrugated core may become adequate alternative for thick traditional plywood boards (>30 mm) in several fields like road and maritime transport demanding reduced weight and sufficient load bearing capacity. However some scientific effort is required to develop functional product with optimal crosssection parameters.

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The convenient option for analysis of the 3D plywood panels is Finite Element Method (FEM) integrated in computer software. Employing of parametrical model in development process allows saving time in design optimisation using metamodelling technique and elaboration of design guidelines. Guidelines could help engineers and end users to find the most appropriate sandwich type satisfying their demands, without using structural design software.

To use this design method detailed parametrical model validated with physical tests is needed. Considering that plywood are modelled as multilayer material consisting of veneers composed in several layers with different orientation of fibres, the mechanical and physical behaviour of laminate is largely dependent upon the performance of each individual material layer and its bonding (Wu et al 2005).

Numerical analysis of 3D corrugated structures have been described in several papers (Hunt 2004, Hudson et al. 2010) where good correlations between experimental and numerical results were found. Sandwich plywood panels with rib-stiffened and corrugated core have been investigated by (Zudrags et al. 2009) with aim to increase plywood specific stiffness. Optimisation procedures using stiffness and weight ratio for plywood sandwich panels with rib-stiffened core described in (Kalnins et al. 2009).

The aim of this paper is to use validated numerical model of plywood sandwich panels to execute optimisation tasks and create design guidelines for favourable panel types.

## MATERIALS AND METHODS

### FEM modelling

For numerical simulation of the bending tests a FEM commercial code ANSYS v.11 (2009) has been applied. Parametrical model of the panel was created with variable cross section parameters and bending loading set up options. Corrugate V-core plywood sandwich panel has been modelled according the EN 789 (2004) test set-up by using ANSYS 4-node shell element SHELL 181. It has been assumed that each ply has thickness of 1.3 mm and transversal isotropic material properties (Fig. 1). Numerical model geometry was created to match the panel dimensions according to manufacturing tolerance where the thickness of outer plies has been reduced by 20%.

Mechanical properties used in numerical model were obtained in the previous study (Labans et al, 2010).



Fig. 1. Finite element model. Deflection shape and stress distribution in sandwich panel layers.

### **Experimental investigation**

Three sandwich panels with corrugated core have been tested in 4-point bending set up according to EN-789 (Figure 2) at the Riga Technical University, Institute of Materials and

Structures (IMS). The average length of the panels is 1200 mm, width -300 mm, and thickness 30 mm, width of one corrugate wave -75 mm. Surfaces plate manufactured of 5-layer plywood and corrugate core from corrugated 4–layer symmetrical plywood sheet. Deflection has been recorded with extensometer at the middle of the span. Strains on outer surfaces measured using strain-gauges (produced by HBM). For two panels strains were measured also in several positions on corrugate core surfaces. In total 14 strain-gauges were used to cover one panel.



Fig. 2. Experimental 4-point bending set-up according EN 789.

### Metamodelling procedure

In industrial applications, in order to reduce the development time involving the high precision simulations, the metamodels also called surrogate models can be constructed to replace the original response with the approximation functions (Kalnins et al., 2009). Design optimization process using metamodels usually consists of three major steps: 1) design of computer experiments 2) construction of approximation functions that best describes the behaviour of the problem 3) employing developed metamodels in optimization task or derivation of the design guidelines. In current research a sequential design based on Means Square error criterion has been evaluated by EdaOpt software (Auziņš et.al 2007). A total of 125 points for four design variables have been evaluated.

Four design variables have been used to describe different cross section parameters, in particular number of plies in upper *P1* and corrugate plates *P4*, the total section height *P2* and the angle between the upper plate and the corrugate core *P3* as displayed in Figure 3. Design boundaries for the variables are given in table 2. As response parameters acquired during the numeric calculations are maximum deflection at the midspan U, normal stress at the midspan  $\sigma$  and the tension strain in outer ply  $\varepsilon$ .



Fig. 3. Variable parameters of cross section for sandwich panels with corrugated core.

### **RESULTS AND DISCUSSIONS**

### Validation of numerical model

To validate the numerical model of he plywood sandwich panel, experimental strain and deflection measurements have been compared with the response values extracted from numerical simulations. The numerical and experimental deflection curves are compared in Figure 4. One can note that load deflection curves have linear behaviour, indicating the elastic deformation of the panels. Numerical results practically match the experimental load/deflection values. Vertical line added to the graph in order to identify the deflection limit state (5% of span length) prescribed by structural safety codes.



**Fig. 4.** Experimentally obtained strain values compared with numerical results (a- longitudinal strains on inner surfaces; b- shear strains on corrugated ply).

Curves with negative strain values are obtained from strain-gauges attached to the upper surfaces of the cover panels or in compressed zone. Load/ strain curves derived from the strain gauges attached at the corrugated core surface are summarized in Figure 8 b). Shear strain values obtained by numerical modelling are higher than experimental values in average by 10-15 %. This could be explained by inaccurate positioning of the strain gauges, because strains should be measured in 45 degrees angle toward the panel longitudinal axis. Precise measuring angle probably was not reached or maximal strains were positioned at different angle because of not precise veneers orientation in plywood sandwich production.

### **Optimisation results**

During optimization procedure the maximum stiffness and volume ratio combinations has been obtained for given parametrical variables and normalized versus homogeneous plywood panel (Table 1). Approximation functions have been elaborated using VariReg software (Jekabsons, 2009) Adaptive Base Function Construction (ABFC) method has been used to create multiorder parametric polynomial approximation functions. Optimal values of variables have been found using approximation functions and MS Excel Solver software.

geometry.							
Variable	Set1	Set 2	Set 3	Set 4	Set 5		
<i>P1</i>	3	5	5	3	3		
<i>P2</i>	0.03	0.035	0.04	0.045	0.05		
<b>P3</b>	5	5	3	5	5		
<i>P4</i>	60	60	60	60	60		
$Vs, cm^3$	3966	6600	5580	5300	5360		
<i>Vs</i> - <i>Vp</i> ,%	55	37	53	61	64		
<i>Us-Up</i> , %	22	14	30	35	42		
Total, %	33	23	23	26	22		

**Table 1.** Optimal plywood sandwich parameters sorted according to homogeneous panel

Sandwich panel volume parameter has been marked as Vs in contrary to homogenous plywood volume as Vp. Respectively Us and Up – deflections for sandwich panels and plywood panels of the same thickness. To estimate efficiency of cross section parameters deflections and volumes of sandwich panels were compared with homogenous plywood values. Difference between sandwich panel and pure plywood volume has been divided by sandwich panel volume to acquire volume reduction (%) using sandwich structure. Similar action has been to asses the deflection values for both structural types. The parameter **Total** stands for difference between volume gain and deflection loss (%). In average a total gain value in comparison with homogenous plywood is 25 %. For the sandwich panel of each thickness with optimal cross section parameters design guidelines were evaluated for better demonstration of load bearing capacity at different span length (Fig. 5). One may notice that the load bearing capacity decreases exponentially by increasing of the span length. For other panels the span/ limit load graphs have been constructed as well to assess the load carrying possibilities for panels with different cross-section parameters.



**Fig. 5.** a) Acceptable load / deflection graph for panels with 30 mm thickness, 3-layer surfaces and corrugated ply angle  $60^{\circ}$ . b) Load /span length graph for deflections limit 5 % of span length.

### CONCLUSIONS

Within the present investigation of all-plywood sandwich panels with corrugated core, a multilayer numerical model with variable cross section parameters have been evaluated and verified with the experimental results. A good agreement between the experimental and the numerical results has been acquired. Discrepancy between experimental and numerical results not exceeds 15 %, which is an outstanding result considering wide discrepancy of wood material. It has been concluded that the corrugated panel stiffness is largely dependent on the

corrugated ply angle. The best results acquired from the optimisation procedure indicated the  $60^{\circ}$  corrugate plate angle, however, this value is mainly influenced by manufacturing restraints. The optimisation results demonstrate that in some combinations of design variables the sandwich panels could be up to 40 % weight effective comparing with homogeneous plywood panels with the corresponding height, by losing only 10-20 % of the load carrying capacity. Based on the optimisation results the design guidelines were constructed for a limited amount of considered panel configurations delivering the optimal cross section parameters, within the given deflection limits.

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# MODIFICATION OF WOOD WITH AN ANTI-OXIDANT TANNIN DERIVATIVE: PRELIMINARY STUDY

# Sidorova, E.<sup>1</sup>, Trey, S.M.<sup>2</sup> & Englund, F.<sup>3</sup>

### ABSTRACT

In this study it is investigated if the anti-oxidant propyl gallate, a derivative of tannins, can be used to impregnate wood, react with wood, and provide increased resistance to UV degradation and fungal attack. Samples were vacuum-pressure impregnated with three chemical solutions all based on solvent methanol:water 1:1: 1. Arquad (0.1 wt%); 2. Propyl gallate (10 wt%); 3. Propyl gallate (10 wt%) and arquad (0.1 wt%). Reference samples were impregnated with just solvent methanol:water 1:1. After impregnation, half of the samples were vacuum treated and the other half were oven-treated. The samples were then investigated in terms of reaction of propyl gallate with wood by Fourier-Transform Infrared (FT-IR), Thermal gravimetric analysis (TGA), and dynamic scanning calorimetry (DSC). Further studies will include their weight percent gain (WPG) and leaching measurements with examination of the resistance of the samples to fungal attack using soil box testing (ENV 807) and UV durability by QUV advanced weathering and a colorimeter.

Keywords: metal free protection, modified wood, fungicide

Key words: anti-oxidant tannin, wood modification

### INTRODUCTION

Resistance to fungal degradation is one of the most important properties of wooden products intended for applications with outdoor exposure. Chemical modification is an effective way to improve fungal resistance. Due to the pressure from environmental requirements, there is a constant demand in investigating new organic chemicals for wood preservation.

The aim of the study is to investigate a non-metal organic fungicide propyl gallate, and to determine if it can improve the properties of wood. Propyl gallate is an antioxidant derived from gallic acid contained in tannins of wood. Thus it is a natural anti-oxidant that may provide fungal durability. Wood has trace metals which different types of fungi need in order to grow and break down wood. Propyl gallate has antimicrobial activity and has been shown to be an effective chelating agent or binder of trace metals. [Binbuga 2005, Baldrian 2002] Brown rot fungi are known for generating hydroxyl radicals which breakdown lignocelluloses. [Backa 1992, Irbe 2011] Propyl gallate has radical scavenging ability [Perron 2008] and thus may prevent lignocelluloses breakdown in case of brown rot.

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In this preliminary study the results from propyl gallate modified wood are compared to quaternary ammonium additive modified wood and combined with it to determine if synergistic effects are possible. Arquad is the commercial name of the biocide/fungicide. Quaternary ammonium fungicides are known for antimicrobial and disinfecting activity, they are low in toxicity and biodegradable. [Tsunoda 1987, Akzo Nobel 2006]

### MATERIALS AND METHODS

#### Materials

Propyl gallate and methanol were supplied by Sigma Aldrich (Sweden). Arquad 10-50 was supplied by Akzo Nobel, Sweden. All chemicals and solvents were used as received. Scots pine veneers (density 450 kg/m<sup>3</sup>) were supplied by Faner Fabriken (Sweden) and were conditioned at 65% RH at room temperature, resulting in about 10 wt% moisture content. Scots pine for blocks (density 450 kg/m<sup>3</sup>) was supplied by Hallsjö Brädgård AB (Sweden).

### Methods

Scots pine samples of two types were prepared: blocks of 15x25x50 mm and veneers of 1x10x100. Three chemical solutions based on solvent methanol:water 1:1 were prepared for impregnation of wood samples: 1. Arquad (0.1 wt%); 2. Propyl gallate (10 wt%); 3. Propyl gallate (10 wt%) and arquad (0.1 wt%). Reference samples were impregnated with just solvent methanol:water 1:1. 10 blocks and 4 veneers for each impregnation solution were prepared. Blocks were prepared for the further studies of weight percent gain and UV-resistance.

Vacuum pressure impregnation was used (pressure 10 bar, 1 MPa). Before chemical solutions were added vacuum was applied for 20 min. The samples were pressure impregnated for 2 hours. After impregnation half of samples was vacuum treated, another half oven treated in order to determine if propyl gallate would react with wood covalently just by taking away water (vacuum) or if heat was necessary. For the oven-treatment samples were wrapped in aluminum foil and heated at 90°C for 24 hours and then unwrapped and heated further to 120°C in an effort to react propyl gallate to the hydroxyl groups of wood by transesterification. Usually equilibrium reaction is possible by pulling off one of the products, in this case propanol when propyl gallate is reacted with hydroxyl groups of wood (fig. 1). The names of samples are presented in table 1.



Fig.	1.	Reaction	of	propyl	gallate	with	wood.
<u> </u>							

Table 1. Sample naming scheme.						
Impregnation Solution	Treatment after impregnation					
	Vacuum treated	Oven treated				
Just solvent (methanol:water 1:1)	WS_V	WS_O				
Arquad (0,1%)	A_V	A_O				
methanol:water 1:1						
Propyl gallate (10%) methanol:water 1:1	PG_V	PG_O				
Propyl gallate (10%),	PG-A_V	PG-A_O				
arquad (0,1%)						
and methanol:water 1:1						

Table 1. Sample naming scheme.

After impregnation and drying/curing, the veneer samples were investigated in terms of reaction of propyl gallate with wood by Fourier Transform- Infrared (FT-IR), thermal gravimetric analysis (TGA) and dynamic scanning calorimetry (DSC)).

### RESULTS

The results of reaction of propyl gallate with wood (by Fourier Transform- Infrared (FT-IR) are presented in fig. 2. At 1310 cm<sup>-1</sup> it is observed that the in the propyl gallate impregnated sample that was vacuum dried, there is the presence of the the phenol groups at 3300-3500 cm<sup>-1</sup> and a large peak at 1310 cm<sup>-1</sup> from the  $-CH_3$  methyl group of propyl gallate indicating attachment of propyl gallate to wood by transesterification has not taken place to a large extent. However, in the propyl gallate impregnated sample that transesterification has taken place to some degree as ethanol leaves and the carbonyl attaches to the hydroxyl groups of wood. However, disappearance of the phenol groups also takes place as seen by the disappearance of peaks at 3300-3500 cm<sup>-1</sup>, indicating that anti-oxidant character of the available phenol groups may no longer be available to a large extent.

In the fig. 3 the first heat is shown of the DSC. It is observed that propyl gallate has a melt temperature  $(T_m)$  around 150°C. The untreated wood dried vacuum shows a transition at blank temperature and a later temperature when oven dried. The presence of propyl gallate seems to shift this transition to a lower temperature, but the crystalline peak of propyl gallate is not observed indicating that the roughly 13 wt% that is in the wood is well dispersed in the wood and is not able to organize to crystallize with itself.



Fig. 3. DSC diagram

In fig. 4 the TGA plot is presented. It is observed that propyl gallate does not begin to degrade or volatilize until around 300°C. However, all veneers begin to degrade around the same temperature indicating little improvement in the thermal stability at high temperatures with the addition of the anti-oxidant propyl gallate. Further, veneers that have been treated with propyl gallate lose less mass before 130 °C, which could be water and bound water, indicating that the treated veneers are more hydrophobic and could have better resistance against water sensitivity.



### CONCLUSIONS

The propyl gallate and an organic fungicide were impregnated in veneers in an effort to use the anti-oxidant properties of propyl gallate for anti-fungal protection. The propyl gallate was observed by FT-IR to not react to wood until heated in the oven and then the disappearance of the phenol groups was observed. The propyl gallate was observed to be well dispersed in wood by DSC and the lack of crystallinity of the propyl gallate. Further, the wood was more hydrophobic as observed by TGA, but maintained the same degradation temperature with and without treatment. Weight percent gain, dimensional stability, QUV, and soil box testing results will be presented later in order to determine if the addition of propyl gallate has an effect and if the addition of an organic fungicide is synergistic.

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# WET STORAGE OF BIRCH LOGS FOR PROTECTION AGAINST DISCOLOURATION

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### ABSTRACT

Discolouration ("bränning" in Swedish) is a well-known problem that can occur if birch logs (*Betula sp.*) are stored outdoors for more than a few weeks during the summer. The risk of discolouration, combined with the considerable need for storage during the summer owing to the uneven wood supply and the closing of mills for vacation, represents a substantial problem for Swedish sawmills specialising in birch. Traditionally, wet storage (i.e., irrigation or sprinkling) has not been used in Sweden to protect birch logs. The documented knowledge of the wet storage of birch is very limited compared with the detailed knowledge of softwood wet storage. Therefore, a storage study was performed in southern Sweden to compare the end-surface discolouration of wet- and dry-stored birch during 12 weeks of summer storage. Two full-sized experimental piles of roundwood were set up at a log yard. One of the piles was properly water sprinkled during the entire period, whereas the other pile was dry stored. The logs from the piles were compared to determine the potential of wet storage for protecting stored birch. The results showed that wet storage significantly reduced discolouration in comparison with the discolouration observed in dry-stored logs.

Key words: stain, *Betula sp.*, wood storage, irrigation, sprinkling.

### INTRODUCTION

The interest in birch (*Betula pubescens* and *Betula pendula*) saw logs and pulpwood is currently high among forest owners, the forest industry, and its customers in Sweden. However, knowledge of the techniques for handling birch wood from forest to industry is not as satisfactory as the corresponding knowledge for spruce (*Picea abies*) and pine (*Pinus sylvestris*), the two most common commercial forest tree species in Sweden. In order to make the use of birch wood more attractive, additional knowledge about such subjects as storage is desirable. Methods for the storage of spruce and pine are well known, and wet storage (i.e., irrigation or sprinkling) is the common method for protecting stored roundwood (e.g., Staland et al. 2002, Liukko 1997). Very few publications have discussed the outdoor storage of birch under the conditions found in Sweden (e.g., Corbo et al. 2001, Ståhl et al. 2005). To the best of my knowledge, the wet storage of birch has not yet been investigated.

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The principal problem affecting birch stored at sawmills during the summer is the brownish discolouration starting at end surfaces (stain or "bränning" in Swedish), which reduces the quality of the logs. The discolouration spreads from the end surfaces of the logs into the logs themselves and causes economic losses for the industry. The strength of the wood is not affected, but the quality of the discoloured boards is strongly affected. It is probable that the discolouration is caused by oxidation that occurs when air penetrates the wood. The mechanisms underlying the discolouration are, however, not fully explained (Corbo et al. 2001, Ståhl et al. 2005). This type of discolouration in stored birch only occurs in conjunction with the slow drying of logs outdoors. The controlled drying of sawed goods is not affected. The discolouration can grow rapidly, affecting several hundred millimetres of surface area in only a few weeks (Nylinder et al. 2007). Nylinder et al. (2001) conclude that the storage of hardwoods during summer can be very troublesome if discolouration penetrates deeply into the log and damages a large volume of wood. This discolouration can be difficult to distinguish from the red heart that is common in birch and described by for example Drouin et al (2010). The discolouration discussed in this study starts after felling contrary to the red heart.

Drying, cracks and deterioration of birch can also be a problem during storage, especially because storage periods can last up to several months. These long periods result from harvesting conditions in the forest that produce an uneven wood supply for the industry. They are also a consequence of the closing of mills during vacation (commonly four to five weeks starting from the beginning of July). It is reported that birch is more heavily degraded by rot fungi during storage in the forest than spruce and pine (Tamminen 1979). Clearly, it would be valuable to find a way to minimise problems of this type during the storage of birch, just as they can be minimised with wet storage during the storage of spruce and pine.

It is known that snow or cold storage and sealing with plastic sheets effectively protect stored birch saw logs (Corbo et al. 2001). None of these methods (and especially not sealing) are used to any great extent in Sweden today. Wet storage effectively protects spruce and pine from damage during storage, but it is not known how wet storage would affect stored birch.

The aim of the study was to investigate the effect of wet storage on discolouration on stored birch saw logs. Birch logs with and without wet storage were compared. The logs were stored for 12 weeks during the summer in southern Sweden. Discolouration was determined at the beginning of storage, after six weeks, and after 12 weeks. Logs harvested during the winter and logs harvested during the spring, when the sap was rising, were both included in the study.

## MATERIALS AND METHODS

The storage site was located at the Södra Interiör sawmill in Traryd, in Markaryds municipality in the Småland region of southern Sweden. The birch logs used in the study were harvested within 200 km of the sawmill. The relative proportions of *Betula pubescens* and *Betula pendula* in the mixture of birch logs used for the study were unknown. However, the birch stands in the area are composed of approximately equal proportions of each species (Nylinder et al. 2001). The logs used in the storage experiment were randomly selected from logs that had arrived at the sawmill. Both logs

harvested during January or February 2011 (winter harvested) and logs harvested during the second half of April 2011 (spring harvested) were used. The experimental logs, 3.2 m long, were placed together with non-experimental aspen and alder logs in two piles, ca. 17 m long and 3 m high. One of the piles was sprinkled 24 h a day with more than 200 mm water per day. The water was recycled water used for sprinkling for the entire log yard (different hardwoods, primarily aspen, were sprinkled). The other pile was dry stored at a safe distance from the closest sprinkler. The logs were stored for 12 weeks, from May 18, 2011 to August 10, 2011. For technical reasons, the wet storage treatment started one week later than the beginning of storage.

Logs were randomly selected from the piles for the determination of discolouration and removed with a crane lorry at the start of storage, after six weeks of storage, and when the storage ended after 12 weeks. At the start of storage, 20 winter-harvested logs and 10 spring-harvested logs were selected. No differentiation was made between logs destined for the different storage treatments. After six weeks, 10 winter-harvested and five spring-harvested logs were selected from each of the two piles (sprinkled and non-sprinkled). At the end of storage, 20 winter-harvested logs and 10 spring-harvested logs were selected from each of the two piles (sprinkled and non-sprinkled). At the end of storage, 20 winter-harvested logs and 10 spring-harvested logs were selected from each of the two piles.

Each log selected was split longitudinally with a chainsaw. The split was made from the top end of the log. The log was split to a length that allowed the measurement of the discolouration. At the end of storage, the logs were cut in half with a mobile Wood-Mizer bandsaw. The discolouration was measured in mm from the top end of the logs and inwards in a longitudinal direction. It was measured both at the base level, where most of the radial surface was discoloured, and at the level that the single deepest discolouration, often visible as a flame-shaped mark, had reached. If no flame-shaped marks were present, the base level and the deepest level (the "flame level") were the same. Discolouration clearly associated with wood damage were omitted. Mean values, standard deviations and t-tests for the data on discolouration were calculated in Microsoft Office Excel 2003.

### RESULTS

The winter- and spring-harvested logs showed limited amounts of discolouration at the start of storage (Fig. 1, Fig. 2). The development of discolouration during the 12 weeks of dry and wet storage is shown in Fig. 1 (base level) and Fig. 2 (flame level).

The differences seen after 12 weeks of storage between wet- and dry-stored logs were significant (p<0.001) for both the winter- and the spring-harvested logs and for both the base level and the flame level. The differences between the storage treatments after six weeks of storage were not significant (p>0.05) for the spring-harvested birch, whereas the differences between wet and dry storage for winter-harvested birch were significant (p=0.036 for the base level and p<0.001 for the flame level). The winter-harvested logs and the spring-harvested logs did not differ significantly (p>0.05).



**Fig. 1.** Base level with standard deviation of discolouration in winter- and springharvested birch during 12 weeks of wet (sprinkled) or dry summer storage, measured in mm from the top end of the logs. n=20 for winter-harvested logs at 0 and 12 weeks, n=10 for winter-harvested logs after 6 weeks, n=10 for spring-harvested logs at 0 and 12 weeks and n=5 for spring- harvested logs after 6 weeks.



**Fig. 2.** Flame level (deepest level) with standard deviation of discolouration in winterand spring- harvested birch during 12 weeks of wet (sprinkled) or dry summer storage, measured in mm from the top end of the log. n=20 for winter-harvested logs at 0 and 12 weeks, n=10 for winter-harvested logs after 6 weeks, n=10 for spring-harvested logs at 0 and 12 weeks and n=5 for spring-harvested logs after 6 weeks.

In addition to the discolouration shown in Fig. 1 and Fig. 2, discolouration also entered the logs at places where the bark was damaged. The growth of the discolouration in the radial direction was very limited compared with the growth in the longitudinal direction. It was also observed that discolouration spread easily in damaged areas, such as cracks and areas of rot in the end surface. At the end of storage, whether the bark was damaged or intact, discolouration could be seen just inside the bark, along the whole log and up to 20-30 mm deep at most. This form of discolouration was more frequent in the drystored logs than in the wet-stored logs for both the winter- and the spring-harvested logs.

## DISCUSSION

The results of this study showed that the discolouration, starting from the end surfaces and growing into the logs, were significantly less for wet-stored birch logs than for drystored birch logs. The growth of discolouration was, however, not completely avoided in the wet-stored logs. It is possible that the wet storage reduced discolouration by limiting the amount of air that could penetrate the wood. The discolouration that could be seen near damaged wood and near the bark in this study further supports the hypothesis that the penetration of air into the wood is of great importance for the occurrence of discolouration.

A hypothesis that spring-harvested logs, owing to e.g., nutritious rising sap, could be more sensitive to discolouration than winter-harvested logs could not be statistically confirmed in this study even if tendencies were seen. The type of storage and the duration of storage were relatively much more important. It should also be noted that greater uncertainty was associated with the spring-harvested birch because fewer spring-harvested logs than winter-harvested logs were used for the experiment. The smaller number of spring-harvested logs resulted from very poor spring logging conditions. Another source of uncertainty was the unknown distribution of *Betula pubescens* and *Betula pendula* in the material. Verkasalo (1993) reported that *Betula pendula* showed longer discolouration (stain) compared with *Betula pubescens* for logs stored in the forest.

The logs already showed discolouration at the start of storage because the storage experiment started after the harvesting date. Moreover, the wet-storage treatment did not begin until one week after the start of storage. These initial conditions were not ideal. However, considering the discolouration after six and 12 weeks, the importance of the initial conditions seems to have been very slight. However, it is possible that different results would have been obtained if entirely fresh logs with no discolouration had been used. Further studies on logs with no initial discolouration are needed. Starting a wet storage study as soon as the air temperature rises above zero would also be interesting. The limited sample sizes used in this study also suggest a need for further, more extensive studies.

Twelve weeks of storage is a relatively long time, but this storage period is realistic for Swedish sawmills owing to variations in the wood supply and vacations, for example. Traditionally, wet storage is not used for the protection of birch in Sweden, but the results of this study suggest that it could possibly be used if the losses owing to discolouration during storage are considerable. In addition to providing protection
against discolouration, wet storage prevents drying and cracking, and the resulting moister wood facilitates processing. The disadvantages of the method are the costs of equipment and labour, the need for access to water and the requirement for permission for wet storage. It is also possible that long-term wet storage of birch can make the wood vulnerable to bacterial damage, as is the case for spruce and pine.

#### CONCLUSIONS

The wet storage of birch logs significantly reduced discolouration (staining) compared with that observed for dry-stored logs in this study. No differences in discolouration were found between the winter-harvested and spring-harvested logs. If storage of birch logs is necessary for an extended period during the summer, wet storage can be one way to reduce the extent of the damage caused by discolouration.

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# THE USE OF ESTERIFIED LIGNIN FOR SYNTHESIS OF DURABLE COMPOSITES

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## ABSTRACT

Lignin is a natural polymer and one of the most abundant materials on earth. Despite this fact, lignin is often viewed as a by-product in chemical pulp processing and the use of lignin as a sustainable material is low. However, research and public awareness of sustainability have opened up new possibilities for using lignin as a material.

In this study, Kraft softwood lignin was reacted with acetic and propionic anhydride to synthesize esterified lignin. Reactions were performed at 80 °C. The product was analyzed using IR, DSC and processability parameters, such as melt viscosity. The product was then blended with LDPE and extruded into composite films. The films were analyzed using FTIR and TGA, and the mechanical properties were determined by tensile tests. Preliminary results are positive and further studies are planned to confirm the results and establish the range of properties attainable.

Key words: Modified lignin, LDPE, WPC, compatibility.

## INTRODUCTION

Lignin is a natural polymer which together with hemicelluloses performs as a matrix material bonding cellulosic fibers together, in order to give strength to the plant cell wall. Considering the fact that it is the second most abundant organic compound on earth, lignin is without doubt an underutilized resource (Fox and McDonald 2005). It is often viewed as a mere by-product from chemical pulp processing and is mainly used for energy generation. However, research and public awareness of sustainable materials have created new opportunities for using lignin. One drawback of using lignin is its low compatibility with common plastics, such as LDPE, but by introducing new functional groups this could be overcome. One method of tailoring lignin into a usable thermoplastic for composites, could be to modify the lignin polymer by esterification. This could reduce the volumes of petroleum based plastics used today and would also introduce a biomaterial to be used in a number of plastic products.

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## MATERIAL AND METHODS

#### Synthesis and production of sample films

In this study, kraft softwood lignin (KSL), kindly supplied by Innventia, was reacted with acetic and propionic anhydride to synthesize esterified lignin (LAP). During the first part of the study, smaller batches using 10 g of KSL, 100 mmol of acetic anhydride and 300 mmol of propionic anhydride, were synthesized. The reactions were performed at 80 °C and 100 °C, in order to determine any temperature effects. For the second part of the study, the reaction performed at 80 °C was upscaled to 100 g of KSL with a corresponding increase of anhydride. The reaction procedure was similar to previously reported ones (Steward et al. 2002). The product was analyzed using FTIR, optical microscopy and processability parameters. The product from the larger batch was then blended with LDPE and compounded into composites using different processing temperatures.

All films were produced by compounding and film extrusion on a DSM Xplore, 15 ml micro compounder / extruder into 30 mm wide films. The dry weight ratio of the LAP was kept constant at 10% for all the samples. Four composite samples were prepared, the first three samples were prepared by dry blending 11 g, compounding and extruding films, the screw speed was kept constant at 50 rpm, the temperature was 150, 160 and 170 °C for the three first samples. The fourth sample was prepared by pre-compounding an 8g, 50/50 mix of LAP and LDPE with a screw speed of 150 rpm and 150 °C. The compound was then dry blended with LDPE to get an 11g blend with 10% LAP followed by compounding and film extruding at the same speed as the first three samples at a temperature of 150 °C (Preblend 150 °C). As a reference, pure LDPE was extruded at 160 °C.

#### **Physiochemical Characterization**

Attenuated Total Reflection-Fourier Transform Infrared Spectroscopy (ATR-FTIR) was used and all measurements were performed in air at room temperature. The equipment used was a Perkin-Elmer Spectrum 2000 FT-IR equipped with a MKII Golden Gate, Single Reflection ATR System from Specac Ltd., London, U.K. The ATR crystal was a MKII heated Diamond 45° ATR Top Plate.

Thermogravimetric analysis (TGA) was used to study the thermal properties of the films. The equipment used was a Mettler Toledo thermogravimetric analyser with a sample robot, and all samples were heated from 25 to 600°C in air.

Differential Scanning Calorimetry (DSC) was used to analyze the thermal properties of the films. The analyses were performed on a DSC 820 equipped with a sample robot and a cryocooler (Mettler Toledo). The DSC runs were carried out in closed sample pans sealed in air using the following temperature program: heating from 25 to 160°C (or 300°C) at 50°C/min, followed by cooling from 160°C (or 300°C) to -20°C at - 50°C/min and then a final heating to 180°C (or 300°C). Before and after cooling the samples were kept isothermally for 10 and 5 minutes respectively.

For the optical microscopic images, an Olympus BX51 was used and the samples were pressed together by glass microscope slides.

Tensile tests were performed on dumbbell shaped samples from all the extruded films at a pulling speed of 10 mm/minute. The width of all samples was 0.3175 mm with a thickness ranging from approximately 0.13 to 0.45 mm.

## **RESULT AND DISCUSSION**

Initially, the produced films were visually assessed and it was concluded that the process temperature does affect the final film (Fig. 1). A starting temperature of 160°C lead to a brown colored film, indicating well-distributed LAP in the LDPE, and an increase in temperature to 170°C resulted in a highly separated film, with small islands of aggregated LAP in the LDPE matrix. However, a decrease in temperature and preblending of the two components resulted in an improvement of the produced films, which was also supported by the microscopic images (Fig. 2).



**Fig. 6** Photograph of films. From left: Pure LDPE, LAP 150°C, LAP 160°C, LAP 170°C and LAP 150°C pre-blend.



Fig. 7 Microscopic images of the produced films.

By studying the FTIR spectra (Fig. 3) of the unmodified and the esterified lignin (LAP) it was confirmed that the reactions had occurred since some large differences could be seen in the spectra. One of the most obvious differences was the major decrease in the peak at approximately  $3000-3600 \text{ cm}^{-1}$ , representing the phenolic and aliphatic hydroxyl groups. Another difference is the large increase of the carbonyl peak at *ca* 1730 cm<sup>-1</sup>, which originates from the esters formed when the anhydrides react with lignin hydroxyl groups. Both of these differences are in accordance with previous studies (Steward et al. 2002, Glasser and Jain 1993).



**Fig. 8** FTIR spectra of lignin (black line) and modified lignin (grey line). The zoomed insertion shows the region of 1900-1300 cm<sup>-1</sup>.

The DSC results (Fig. 4) show a clear melting point  $(T_m)$  for LDPE at 110-120°C. However, the effect of the lignin on the  $T_m$  appears to be very low, and the DSC curves still exhibit a high level of crystallinity, indicating that the miscibility is not that good.



Fig. 9 DSC thermograms (2<sup>nd</sup> heating scan) for lignin, LDPE and two of the four LAPs.

Fig. 5 show the results from the TGA measurements, and by comparing the thermograms for lignin and modified lignin it can be concluded that esterification of the lignin affects the thermal stability of the material. The results also show that the final degradation temperature of the lignin is somewhat increased when modified with anhydride. The curves representing pure LDPE and LAP indicate that adding lignin

could possibly decrease the amount of oxidation during degradation, which in turn increases the thermal stability.



Fig. 10 TGA thermograms for lignin, LDPE and two of the four LAPs.

Results from the tensile testing shows decreased strength for all composite samples compared to the pure LDPE, the 170 °C sample show lower stress at break which is most likely related to large LAP aggregates formed within the composite at this temperature. The stiffness is also lower for the 150 °C, 160 °C and pre-blend 150 °C samples, whereas the 170 °C samples show increased stiffness due to the large aggregates of LAP in the LDPE matrix.



Fig. 11 The graphs illustrate tensile stress at break and modulus for a pure LDPE film in comparison to the four different LAP-LDPE composites.

## CONCLUDING REMARKS

- The FTIR spectra show that esterification of lignin is possible
- The LAP used did not blend well with the LDPE and is in this case to be considered as a filler material
- At 170 °C processing temperature the LAP formed larger aggregates which caused uneven films and crack initiations when mechanically tested

## FUTURE WORK

Future work of interest is to use modified lignin that has been esterified at 100  $^{\circ}$ C instead of 80  $^{\circ}$ C. The first part of this study indicates that increasing the temperature results in higher degree of acetylation which in turn could increase the compatibility with LDPE.

Another interesting aspect would be to vary the molar ratio between the two anhydrides, and also to use the anhydrides separately.

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## SELF-BINDING FIBERBOARD MADE OF STEAM EXPLODED WOOD: THE CASE OF MEDIUM DENSITY

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## ABSTRACT

The present study is aimed at obtaining self-binding (without any additional binders) medium density board samples from steam-exploded grey alder (*alnus incana* (L.) Moench) chips. The chips used for steam explosion pre-treatment (SEP) were fractionated by crushing to different size from 0.4 mm to 20 mm. The SEP was done in a batch reactor at constant temperature and pressure (235 °C and 3.2 MPa) varying the time from 0.5 to 3 minutes. The pre-treated fibrous mass was air dried to different water content and hot-pressed under various conditions part of the fibrous samples being additionally milled before hot-pressing. The pressing temperature of boards was set at 150 - 170 °C, the pressure – at 0.4 - 2.1 MPa for 6 - 8 min during the hot-pressing and for 10 - 15 min during the cooling of the press plates to 80 °C. The density range of the produced boards is 0.5 - 0.8 g cm<sup>-3</sup>.

To find the effect of the used pre-treatments and of the pressing conditions on the boards various physical properties were tested according to the relevant EN standards. Except for thickness swelling varying from 6 to 19 % regardless of the density, all the other determined properties are found to improve with increasing the board density. Contrary to expectations the tested mechanical properties do not meet the requirements of EN standards.

Key words: grey alder, steam explosion treatment, fibreboard, medium density, mechanical properties.

## INTRODUCTION

The thermo-mechanical pre-treatment called steam explosion has a sizeable potential in different kinds of biomass refineries over the world (Gravitis 1987). By means of that pre-treatment, as it is well known, without addition of any chemicals, the biomass (e.g. wood) structures are easily broken to obtain valuable fibrous lignocellulosic mass (Mason 1926). A lot of studies done with different kinds of biomass confirm the high potential of the mass pre-treated by SE for applications in different fields (Kokta and Ahmed 1998, Gravitis and Abolins 2007, Taherzadeh and Karimi 2008) the high-density boards made of SEP fibres without synthetic adhesives being one of them. The

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studies have been made with boards of different kinds of the raw material, e.g. wood or residues of agricultural species (Laemsak and Okuma 2000, Angles et al. 2001, Velasquez et al. 2003, Van Dam et al. 2004, Tupciauskas et al. 2009). Unfortunately, there are not many reports about medium density fibreboard (MDF) obtained without addition of adhesives. Xu et al. (2003) published medium density board from kenaf (*Hibiscus cannabinus* L.) chippings (<0.25 - >2 mm) hot pressed with steam injection. The board showed promising properties which partially met relative standards. Grinding the kenaf core to smooth particles (53 µm) leads to excellent board strength obtained without any pre-treatment (Okuda and Sato 2004). However, compared to requirements of standards, the form stability of the board needed improvement.

The objective of our research was to obtain self-binding medium density board from locally grown alder wood (*Alnus incana* (L.) Moench) pre-treated by the SE process and hot pressed under various conditions and testing of board properties to find dependence of the properties on manufacturing conditions and to compare the properties with the requirements of commercial MDF standards.

### MATERIAL AND METHODS

The fibreboard samples were obtained from grey alder (*Alnus incana* (L.) Moench) – a fast-growing tree species in Latvia. The raw material was crushed by a "Bruks" (Sweden) wood chipping machine, cleaned of bark and air-dried to constant mass (8-9 % of moisture content). Part of the chips used in SEP had dimensions of ~20 mm (in width), part was crushed by "Retsch" through sieve of 2 mm openings while chippings of size less than 0.4 mm were additionally screened off the fraction.

The SEP for above described materials was done with saturated steam in a batch reactor (Tupciauskas et al. 2009) at constant temperature and vapour pressure (235 °C and 3.2 MPa) at duration 0.5, 1, 2, and 3 minutes. Severity factor  $R_0$  of the SEP conditions was used to present the treatment temperature and time by a single variable. The  $R_0$  expression is shown in Eq. 1 (Overend and Chornet 1987). The exploded fibrous mass was picked up and air-dried to different water content (see Table 1.).

$$R_0 = t \cdot \exp\frac{(T - 100)}{14.75},\tag{1}$$

where 't' is the treatment time in min;

'T' is a temperature of the treatment.

The exploded mass of ~20 mm chips after drying was additionally crushed by "Retsch" through sieve with 2 mm openings (see samples 7 - 9 in Table 1.). To find the effect of homogeneity of the exploded mass one sample was prepared from exploded chippings additionally crushed after the SEP (see sample 10 in Table 1.). To understand the effect of hemicelluloses another sample was washed (W) with water (1:4) after the SEP and additionally crushed through sieve with 2 mm openings (see sample 9 in Table 1.). Two samples (4 and 11) were prepared to detect the effect of water content on board properties (see Table 1.). All pre-treatment and pressing conditions of the samples are summarised in Table 1.

Sample	Chips	SEP	Mass pre-	Water		Pressing	conditions	
Nr.	size for	time,	treatment	content				
	SEP,	min /	after SEP	before	Τ,	p <sub>max/</sub>	$\tau_{\rm pr+cool}$ ,	h,
	mm	$logR_0$		pressing,	°C	p <sub>min</sub> ,	min	mm
		values		%		MPa		
1	0.4 - 2	0.5/3.67	-	8.4	160	0.6/0.05	8+10	8
2	0.4 - 2	1/3.97	-	7.3	150	0.8/0.08	8+15	8
3	0.4 - 2	1/3.97	-	8.6	160	1.5/0.02	8+12	8
4	0.4 - 2	1/3.97	-	9.1	170	2.1/0.02	6+10	8
5	0.4 - 2	2/4.28	-	6.3	160	0.6/0.05	8+10	8
6	0.4 - 2	3/4.45	-	7.4	160	0.4/0.06	8+10	8
7	~ 20	1/3.97	crushed	8.2	170	0.5/0.02	6+10	8
8	~ 20	1/3.97	crushed	8.2	170	0.5/0.01	6+10	6
9	~ 20	1/3.97	W+crushed	15.3	170	0.8/0.01	6+10	6
10	0.4 - 2	1/3.97	crushed	12.7	170	0.6/0.01	6+10	6
11	$\leq 2$	1/3.97	_	19.2	170	0.3/0.01	6+10	6

Table 1. Samples pre-treatment and pressing conditions

The weight of mass with certain water content  $(m_w)$  for pressing was prepared with account for water content  $(W_{rel})$ , board density  $(\rho_b)$  and board volume  $(V_b)$  according to expression:

$$m_w = m_s \, \frac{100}{(100 - W_{rel})},\tag{2}$$

where 'm<sub>s</sub>' is a solid mass weight ( $m_s = \rho_b \cdot V_b$ ).

The first samples (2, 3 and 4 in Table 1.) were designed of the size of 200 x 200 x 6 mm and five replications were made for each pressing regime. Samples 1, 5, and 6 were designed to be of the size 470 x 470 x 6 mm and the size of the rest (7 - 11) was 420 x 420 x 6 mm all with a single replication. The board samples were pressed in a singlestage "JOOS" pressing machine (Germany) with installed programming software. The weighted mass samples put to the frame with inside dimensions notified above (the frame depth -40 mm) and pre-pressed by hand. Then the frame was taken off and the pressing cycle switched on. The pressing cycle was designed as follows: the heated press plates closing (25 s) – hot pressing (for pressing time see ' $\tau_{pr}$ ' in Table 1.) under the maximum pressure  $(p_{max})$  during one minute the rest of time the pressure being at pmin (Table 1.) - cooling the press plates under the pressure as in previous pressing stage (for cooling time see ' $\tau_{cool}$ ' in Table 1) – the press plates opening. The pressure at the cooling stage (to 80 °C) was held to avoid de-lamination of the board. The pressure was set higher because of the distance between press plates ('h' in Table 1) the actual pressure of the pressing cycle was read from the press monitor. A foil sheet was used to separate sample surface from press plates.

The pressed board samples were cut into various test specimens and conditioned before testing density (EN 323, 1993), thickness swelling and water absorption after 24 h (EN 317, 1993), moisture content (EN 322, 1993), bending strength (modulus of rupture) and modulus of elasticity (EN 310, 1993), and tensile strength perpendicular to the

plane of the board (internal bond, EN 319, 1993). The board properties were tested according to the standards and compared with the European requirements for MDF (EN 622-5, 2009 and EN 622-1, 2003). Mechanical properties of hot-pressed fibreboard samples were tested by a ZWICK/Z100 universal machine for testing material resistance.

#### **RESULTS AND DISCUSSION**

The self-binding medium density board properties are summarized in Table 2 (mean values within the 95% confidence interval) where the last row shows the standard EN 622-5 requirements for boards for use in dry conditions (type MDF). From the table can be seen that thickness (t) of the boards vary from 5.3 to 9.3 mm and correlates with the density ( $\rho$ ) of the boards. The moisture content (MC) of all produced board samples comply with the MDF standard and in average varies from 4 to 6.3%. The thickness swelling (TS) of most boards also satisfies the standard. Only the 9th sample which is made of washed and crushed SE fibre mass does not meet the requirement of the standard on TS. This is an evidence that hemicelluloses modified by SEP help to keep the form stability of the boards in water environment. However a lower content of celluloses is more favourable as observed in samples 5 and 6 having the lowest values of TS because of a higher severity factor of SEP. Velasquez et al. (2003) also approve that the higher severity factors of SEP, the lower content of hemicelluloses remains. The board sample 1 has also a higher value of TS like sample 9 but in this case the lower severity of SEP (logR<sub>0</sub>=3.67) plays the main role.

In Table 2 TS of all other board samples varies from 6 to 10% being very satisfactory values. Comparing samples 4 and 11 differing by water content before pressing a statistically significant (t-Test) difference in TS values is observed. This means that TS of the boards depends on water content of the raw mass.

Nr.	t,	ρ,	MC,	TS,	WA,	MOR,	MOE,	IB,
	mm	g cm <sup>-3</sup>	%	%	%	$N \text{ mm}^{-2}$	$N \text{ mm}^{-2}$	$N \text{ mm}^{-2}$
1	$7.4 \pm 0.03$	$0.63 \pm 0.03$	4.8±1.12	17±4.4	87±9	$2.2 \pm 0.6$	667±157	$0.12 \pm 0.03$
2	$6.2 \pm 0.07$	$0.82 \pm 0.1$	$5.2 \pm 0.14$	$10\pm0.4$	52±12	$4.5 \pm 1.2$	$1252 \pm 247$	$0.27{\pm}0.02$
3	$6.2 \pm 0.04$	$0.78 \pm 0.05$	$4.9{\pm}0.46$	$9\pm0.5$	$57\pm7$	$4.4 \pm 0.9$	$1291\pm351$	$0.28{\scriptstyle\pm0.07}$
4	$6.6 \pm 0.04$	$0.76 \pm 0.04$	$4.8 \pm 0.25$	9±0.3	57±5	$4.9 \pm 1.2$	$1374 \pm 362$	$0.26{\scriptstyle\pm0.04}$
5	$6.8 \pm 0.04$	$0.69{\pm}0.06$	$4.7{\pm}1.29$	6±0.8	69±9	$2.3 \pm 0.7$	734±225	$0.30{\pm}0.07$
6	$7.4 \pm 0.03$	$0.64 \pm 0.06$	$4.0\pm0.9$	6±0.9	$75\pm8$	$1.3\pm0.4$	504±151	$0.19{\pm}0.05$
7	$9.3 \pm 0.17$	$0.48 \pm 0.02$	$6.3\pm0.08$	$7\pm0.5$	143±11	$0.5 \pm 0.08$	$76\pm38$	$0.03{\pm}0.02$
8	6.1±0.19	$0.68 \pm 0.02$	$6.0\pm0.1$	10±1.3	$78\pm8$	$1.9\pm0.2$	434±87	$0.17{\pm}0.01$
9	$6.8 \pm 0.21$	$0.65 \pm 0.03$	$5.1 \pm 0.07$	$19\pm1.5$	$107 \pm 10$	$3.6 \pm 0.7$	$547{\pm}103$	$0.07{\pm}0.03$
10	$5.9{\pm}0.18$	$0.72 \pm 0.02$	$5.4{\pm}0.07$	$8\pm1$	64±5	$5.4 \pm 0.8$	1147±99	$0.25{\scriptstyle\pm0.04}$
11	$5.3 \pm 0.15$	$0.74 \pm 0.03$	$5.8 \pm 0.08$	$7\pm0.8$	$58\pm8$	$3.9 \pm 0.9$	960±225	$0.29{\pm}0.05$
Stand.	> 6 - 9	0.4-0.9	11	17	_	23	2700	0.65

Table 2. Grey alder self-binding fibreboard properties

Water absorption (WA) property of the obtained boards shows similar trends as in case of TS. However, contrary to TS, the boards WA very well correlate with density of the boards as shown in Fig. 1. In this case there is a negative linear correlation meaning that

WA of the boards is lower at higher density. So the TS of the boards depends on SEP conditions, content of hemicelluloses, and content of water before pressing while WA depends on SEP conditions (less than TS), content of hemicelluloses, and the density of the board.



Fig. 1. Self binding fibreboard water absorption vs density.

In spite of form stability, mechanical properties of the boards do not meet the requirements of MDF standard. Probably this is because of insufficient bonding of fibres during pressing of medium density boards under low pressure. Laemsak and Okuma (2000) have observed similar trend of boards from SE oil palm frond fibres. Mechanical properties of the boards with medium density  $(0.7 - 0.9 \text{ g cm}^{-3})$  did not meet the relevant standards. The authors also noted that all tested properties (except of TS) correlate with density of the boards like in our case. Possibly the main problem of self-binding medium density boards is pressing conditions and mass homogeneity. Analyzing the influence of water content of pressing mass on mechanical properties no significant dependence was observed (samples 4 and 11 in Table 2.). Crushing of exploded fibres before pressing neither shows any significant effect on the board properties (samples 4 and 10 in Table 2.). Density seems to have the main effect on mechanical properties of boards while SEP conditions are the next. Washing out of hemicelluloses gave increase of modulus of rupture (MOR) while decreasing the form stability and strength of internal bond (IB) (samples 8 and 9 in Table 2.). In general the optimal SEP conditions for the board properties are at  $logR_0=3.97$  and the optimum pressing conditions are found to be at 170 °C (samples 4 and 10 in Table 2.). The pressure and time do not show a significant effect on the properties of the boards.

#### **CONCLUSIONS**

- The obtained self-binding medium density  $(0.5 0.8 \text{ g cm}^{-3})$  fibreboards from steam exploded grey alder chips show sufficient form stability (thickness swelling and water absorption) but poor mechanical properties (bending and internal bond) compared with requirements of EN 622-5 type MDF.
- Except of thickness swelling, all other tested board properties correlate and improve with increasing the density.

Homogenizing the exploded fibres by crushing did not show significant effects on the tested board properties.

- Increase of water content of the raw mass significantly decreases thickness swelling and the modulus of elasticity while other properties show insignificant differences.
- Washing out the exploded fibrous mass with water increases a bit the modulus of rupture at bending but decrease the form stability and internal bond.
- The optimal producing conditions of the boards are found to be at severity of SEP at  $\log R_0=3.97$  and pressing temperature of 170 °C. The pressure and pressing time do not show significant effect on the tested properties of the boards.

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# IN-SITU POLYMERIZATION OF POLYANILINE IN VENEERS AND THE EFFECT ON MATERIAL CONDUCTIVITY, MORPHOLOGY, AND FLAME RETARDANCE

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## ABSTRACT

Impregnating an aniline monomer solution into wood and reacting polyaniline particles in-situ, in order to take advantage of the complex and robust structure of wood, has been investigated to a lesser degree. Of interest is also how the impregnation and reaction process affects the wood properties in terms of degradation of the material by the acidic impregnation solutions, where the polyaniline particles form within the wood anatomy, what amount of weight percent gains can be achieved, can a conductive veneer be achieved by this approach. In this study, the modification of wood is studied including the effect of the hydrophilic character of polyaniline on the dimensional stability of wood, if the particles leach from the material over time, and if wood becomes more flame retardant with the addition of polyaniline.

Key words: veneer, impregnated, conductive polymer, wood polymer composite

#### INTRODUCTION

Interest of organic conducting materials has been occurring in the past few decades with the most recognition of the area being the 2000 Nobel Prize in chemistry for the development of conductive polymers awarded to Heeger et al. (Heeger 2001, Rimbu et al 2006).

Polyaniline is of interest in many different applications including electronic devices, batteries, solar cells, electromagnetic radiation, anti-static materials, electric heaters, and even filtration of heavy metals (Bhadra 2008) However, conductive polymer have poor mechanical properties, are difficult to dissolving or melting them, and so have been combined with traditional thermoplastics such as a polyethylene terephthalate and polyethylene (Madani et al.). These composites are typically heavy, dense, have weaker matrix physical properties due to the high loading of particles, and shed particles over time leading to contamination.

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The aim of this project is to use the complex structure of wood as a template material that is by nature a light yet strong material and modify with organic conductive polymers in order to obtain a novel functional material. The proposed advantage is not only to obtain a conducting material, but also to address other important restrictions for wood such as the effect of modification on the fungal durability, fire resistance, dimensional stability, strength, and morphology of the conductive polymer within the architecture of the wood structure.

## MATERIAL AND METHODS

The crown cut/flat cut Carolina pine veneer was received from Specialised Veneers Ltd. In Aylesburg, United Kingdom. The aniline, phosphoric acid, sulfuric acid, n-dodecylbenzenesulfonic acid sodium salt, and peroxydisulfate were all used as received from Sigma Aldrich.

Southern Yellow Pine (Carolina Pine) veneers of  $0.6 \ge 5 \ge 5$  mm in sets of 5 were vacuum pressure impregnated for 24 h at 10 bars under inert atmosphere with a chilled 0.2, 0.1, and 0.5 M solution of aniline in water, phosphoric acid (phos) (sulfuric acid (SA), or n-dodecylbenzenesulfonic acid (DBSA)) (dopant)/aniline molar ratio of 1 and peroxydisulfate/aniline ratio of 1.25. Leaching studies were done according to the EN-84 standard.

Cone calorimetry was performed according to ISO 5657 for Ignitability and ISO 9705 for rate of heat release. For these tests the veneers were glued to gypsum boards. Modified veneers were cut of 0.5 cm thickness, l, and 3.0 cm diameter, d, and were placed tightly between two gold plate electrodes connected to a Solartron 1296 electrochemical interface coupled to a Solartron 1260 frequency response analyzer, and the impedance spectrum was measured with an AC amplitude of 10 mV and frequency range from 1 MHz to 0.1 Hz. The resistance at low frequency, R, was collected and analyzed by the SMaRT software, from Solartron Analytical. The conductivity,  $\sigma$ , was then calculated as  $\sigma = \frac{4l}{\pi d^2 \cdot R}$ .

## **RESULTS AND DISCUSSION**

After vacuum pressure impregnation of pine veneers in a monomer solution of aniline and reacting to form polyaniline in the wood structure, it can be observed in Fig. 1 that the weight percent gain of the increases with increasing solution concentration and the dopant does have an effect on the amount of polymer that is taken up by the wood (weight percent gain (WPG)). The leaching after 14 days in water is significant relative to the polymer WPG, but a large degree of conductivity remains. This indicates that a fraction of the leachable compounds comes from the wood itself, possibly due to acid hydrolysis.



Fig. 1. Gravimetric and volumetric determination of polymer WPG and leaching.

By swelling the modified samples in water for 14 days, the dimensional stability could be observed by measuring the difference in the volume swelling and the hydrophilicy was measured by the weight uptake of water. All treatments with polyaniline decreased the hydrophilicity of the samples, with impregnation solution concentration correlating to a reduction in water uptake. The dopant did not have a significant effect on this property. The volume swelling of the modified samples does not appear to be affected by the polyaniline concentration or the dopant used.



**Fig. 2.** Weight of water uptake of the modified wood samples and amount of volumentric swelling or bulking in water after 14 days according to EN-84 standard.

The wood-polyaniline composites showed a degree of semi-conductivity that is not as conductive as polyaniline alone, but shows a conductivity level that is comparable to traditional commercial antistatic materials. This is promising because even with these initial preliminary results, with WPG of less than 10 wt%, it is most likely that aniline goes into the cell wall and polymerizes in the wood connective network and not just within the lumen voids, where it would be isolated and not able to form a conductive material.

	Unleached	Leached in H <sub>2</sub> O 14 days
Sample	$\sigma$ (S cm <sup>-1</sup> )	$\sigma(\text{S cm}^{-1})$
Phos-PA 0.2 M	9.0 x10 <sup>-5</sup>	$2.4 \text{ x} 10^{-6}$
Phos-PA 0.1 M	2.6 x10 <sup>-6</sup>	$1.6 \text{ x} 10^{-8}$
DBSA-PA 0.1 M	8.6 x 10 <sup>-7</sup>	3.7 x 10 <sup>-9</sup>
SA-PA 0.1 M	4.1 x 10 <sup>-5</sup>	$1.3 \times 10^{-8}$
Phos-PA polymer	$3.3 \times 10^{-2}$	
SA-PA polymer	2.0 x 10 <sup>-2</sup>	
DBSA-PA	3.3 x 10 <sup>-3</sup>	
polymer		

**Table 1.** Conductivity of different polyaniline doped polymers and polyaniline veneers

 before and after leaching

It was observed by light microscopy that the adhesion of polyaniline did not occur to a large extent in the lumen. (Fig 3.) By optical light microscopy and by SEM, the lumens were oserved to remain clear of polymer. The polyaniline is highly colored due to the conjugated structure. It will be investigated where in the structure the polyaniline deposited within the cell structure by FT-IR imaging and fluorescence imaging.



Fig. 3. a.) Unmodified vs. b.) Phos-PA 0.1 M unleached.

## CONCLUSIONS

It is possible to obtain veneers that have a range of conductivity by impregnating them with aniline and reacting to form polyaniline in-situ. In addition to the conductivity that allows for these materials to be used for a wide range of applications from anti-static to filtration of waste water, to electromagnetic shielding, the polyaniline has also improved the water sensitivity and flammability of wood.

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# DENSITY VARIATION IN OVER-MATURED LITHUANIAN PINE (*PINUS SYLVESTRIS*) TREE STEM

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## ABSTRACT

The main aim of research was to investigate spatial physical and mechanical properties in over-matured (more than 300 years) pine tree stem. Research completed using two concepts. In the first model, density and stiffness-strength variation was observed by the pith-to-bark and longitudinal direction estimating distance from pith to centre of each specimen in millimeters. In the second approach, number of tree rings estimated in order to evaluate cambium age, eccentricity, and then density variation in radial pith-tobark direction.

Specimens' properties taken from over-matured pine tree butt, middle, and top logs were modeled in spatially polar coordinate system. Variation of results explained by numerical models and visualization in black and grey scale graphical models.

Key words: over-matured wood, density, cambial age, radial-longitudinal variation.

## INTRODUCTION

The density of wood is impact of complex and long processes, based on genetic and environmental conditions that influence on variation of quantity of juvenile, matured and over matured wood in the tree. Significant variation of density begins from 25-60 year (Shawn et al. 2007). Exist opinions, that the density of the same species of tree stem in some cases can differ by 10-20 %. In most species of wood the biggest density is in the bottom of the stem and towards to top the density decreases by about 1.5 % per stem height meter.

At Lithuanian conditions when coniferous species reach approximately one hundred years age the average density develop tendency to diminish (Chan 2011).

Formation of matured wood begins only when tree attain specific age. Therefore, quantity of maturity wood is related to the age but also there is pronounced accent of negative correlation with tree growing speed. Formation of mature wood is such phase-mode changes, when tissue with live cells experience transition to the substance with nearly no metabolism (Karenlampi and Riekkinen 2002).

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Variation of density from toward bark is direct result of the cambium maturing. Overmatured trees often expose observable density reduction close to the bark when measured at DBH and at 60 %-70 % relative stem height (Gryc 2011). In the crosssection in radial direction from bark towards pith, the differences in density can be from 5 % until 20 % or more.

From anatomic aspect, juvenile wood may be described by stepwise variation of dimensions and changes in form, structure and cells distribution in annual rings respectively.

In some cases over-matured wood have linear density decrease toward bark at 50 % of relative stem height and higher. One of the explanations is that extractive materials in pine mature wood twice exceeding that in the sapwood. The highest quantity of extractive materials in the mature cross-section part is in the butt log, where extractive materials distribute almost equally throughout all section length (Gryc 2011).

As can be find in some literature sources decreasing of basic density from bark towards heartwood in radial direction is most visible in fast-growing trees. Conversely, no such features tendencies observed in the slowly and at dense sites growing trees. It can be find narrower rings near the heartwood and at the same time higher densities compared to the sapwood rings together with the ring widening and density decreasing towards the sapwood (Grekin and Verkasalo 2010).

Radial density variation in the stem usually is determined in to ways. When cambium age is evaluated, annual rings from pith towards bark is specified. This in turn impose that specific coordinates are not related to the same calendar year because upwards from bottom annual rings are formatted at different periods. Another way is to calculate annual rings from bark toward wood pith. It means that at different stem heights annual rings were formatted the same year (Jyske et al. 2008).

## MATERIAL AND METHODS

Wood samples cut from over-matured pine (*Pinus silvestris*) stem bottom, middle and top parts used for testing (Fig. 1). Over matured pine tree, sections (about 300 year's old age) selected at different stem heights. Logs were cut from stem and from each log onequarter parts were cut. Bottom part dimensions were ( $2240 \times 700 \times 610$ ) mm, middle part dimensions were ( $2290 \times 500 \times 420$ ) mm and top end part dimensions were ( $1149 \times 410 \times 400$ ) mm respectively. Samples were split into smaller parts from outside (sapwood) towards heartwood direction gradually (Fig. 2). Specimens were cut systematic diminishing their sizes starting from sapwood. Finally,  $65 \times 60$  mm cross-section wood specimens were received.

For investigation of variation of density in radial direction, two methods were used: calculating quantity of annual rings (cambium age) and measuring distance from specimen center towards the pith. Graphical model of the samples, finite specimen dimensions and physical groups of samples are presented in Fig. 3. One –quarter section density variation was modeled considering sawing kerf and at each splitting stage samples were measured with the accuracy ( $\pm 1$  mm). In theoretical model either distance from pith to the specimen center, mm, was fixed drawing lines in polar coordinate system and cambium age was calculated. Cambium age was fixed at the center of every specimen.





- Fig. 1. Pine stem cutting scheme: 1 bottom part samples, 2 middle part samples, 3 top end part samples.
- Fig. 2. Scheme of sample cutting: a) cross form (radial specimens) method [8]; b) major spatial method.

From theoretical model we can see, that average cambium age is about 250 year. By sapwood quantity, we make assumption, that really age of pine is more than 300 years.



Top end part samples **Fig. 3.** Schemes of the quarter-section samples.

In literature we can find, that in the old pine (around 200 years old) latewood amount is about 40 % while in the top of the stem only 20 % of all volume of tree. Therefore, the density must be bigger in the bottom sample group. The basic densities of the samples are marked in grey-color pallet.

The tendency o increasing of the density towards bark was observed only in top end quarter-section sample group. Another sample groups show lower variation of the density (but-end quarter-section sample group), or considerably higher density in the heartwood central part, what contradict with the usual result findings in the literature [Grekin 2010, Molteberg and Hoibo 2007]. The ratio of early wood and latewood within annual ring is highly variable. It depends on wood species and factors influencing width of annual rings (tree age, climate, growing conditions).

In the literature, the most commonly used method is cutting disks from trunk and after that splitting into the small-scale specimens (Molteberg and Hoibo 2007). The main task of our experiments was to ascertain structural specimen sizes to estimate density variation and real influence on stiffness-strength wood properties alongside the tree stem.

Statistical investigations were made using program software "STATISTICA". Linear and non-linear regressions for best modeling adequacy and validation were applied.

## **RESULTS AND DISCUSIONS**

Analysis of the linear regressive equations describing density variations showed that the best correlation coefficient observed in the but-end quarter-section group samples: coefficient of determination  $R^2$ =0.75; for middle group samples coefficient of determination  $R^2$ =0.63 respectively. However, for best-fitted model we must apply nonlinear regressive equations. The biggest score of standard error is for middle group samples 71.88. For bottom and top end group samples score of standard error are 35.59 and 34.92 respectively, similar can be find in the literature sources (Gryc 2011). After comparing calculated and tabulated Fisher criteria it was obtained, that  $F_{calc} < F_{table}$  so models are adequate. Nonlinear regression coefficients are 0.91, 0.9, 0.78 and coefficients of determination are 0.84, 0.8 and 0.62 respectively. Therefore, received nonlinear models adequately describe spatial density variation within over-matured pine trunk.

Variation of the density in the different samples groups in X and Y polar coordinate system (Fig. 2a) applying method (Rikala 2006) given in Fig. 4. As can see bottom and top end stem part group samples values are rather comparable. Density values at middle stem part distribution variate in much larger intervals and are numerically much higher.

Spatial distribution of the density of specimen groups shows the biggest basic density values in the middle stem part samples group (Fig. 5). The median of the basic density in that group is meanly  $100 \text{ kg/m}^3$  bigger than in the bottom and top end parts samples. Obvious decline in density values observed from heartwood towards bark.

Our observations of over-matured pine density variations are contradictory to the regular data about mature trees density properties [Gryc 2011, Karenlampi and Riekkinen 2004) received data was not in line with the experiences that cambial age describes density variations better than distance from the pith (Verkasalo et al., Reynolds 2007). All these phenomena emphasize enormous structural changes in wood tissue when tree reaches 200 and more years.

Variations of the density depending on the cambium age and distance from the pith are presented in Fig. 6 and Fig. 7. As can see in Fig. 6 the density decreases in butt and middle parts of stem after 150 years. Only top end specimens not show great decreasing of the density, when cambium age variety. The biggest values of the density have specimens of the middle part, when reach 120 years. After 150 years the density decreased.



**Fig. 4.** Variation of the density in the stem by X and Y directions.



**Fig. 5.** Variation of the density in the various stem parts.

As can see in Fig. 7 decreasing tendency is lesser. The density of the middle part specimens decreases from 250 mm, while the density values of the butt and top end parts specimen's decreases after 300 mm.



Fig. 6.Variation of the density depending on the cambium age

**Fig. 7**. Variation of the density depending on the distance from the pith

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Some authors (Gryc et al. 2011, Grekin and Verkasalo 2010) affirm, that cambium age must explain variation on the density better than the distance from the pith. However, after our investigations, we can affirm, that variation of the cambium age not explain better the density variation than the distance from the pith.

## CONCLUSIONS

- 1. The density values of middle part of tree stem part are biggest from all investigated quarter-sections and their specimens groups.
- 2. The density of the over matured pinewood is lower near the bark, except of values of top end quarter-section specimens group.

- 3. Decreasing of the density from the butt of the stem towards top end is not uniformly coherent: in the middle section of the stem density markedly increases and then again diminishes towards top-end.
- 4. Distance from the pith to the specimen's center explains density variations better than heartwood specimen cambium age.
- 5. Best-fitted modeling of the density variations received integrating cambium age, distance from pith and average of annual rings per cm at specimen center: for butt part specimens density variation is explained by coefficient of determination  $R^2$ =0.91, for middle part specimens  $R^2$ = 0.90 and top end part specimens  $R^2$ =0.78 respectively.

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# TOOL WEAR EFFECTS ON THE SURFACE FORMATION WHEN MILLING WOOD

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## ABSTRACT

This article presents the research results, showing, how the surface roughness of the wood processed by milling is changing with increase of the cutting path of milling tool. The tests were done with the wood samples of pine and black alder grown in Lithuania. The samples were milled along the fiber in the experimental stand for wood cutting at two different cutting and feed speeds. The roughness parameter  $R_z$  of the processed samples was measured in five sectors using the contact profilometer. The roughness was measured in each sector along and across the fiber. The received measurement results were processed by Gaussian digital filter. The testing results helped to determine that the surface roughness depends on the cutting path, rounding radius of the tool's cutting edge, cutting and feed speeds.

Key words: wood milling, tool wear, surface roughness, pine wood, black alder wood

## INTRODUCTION

One of the main criteria to evaluate the quality of the processed surface is its roughness. It determines the further processing and finish of the surface, esthetical view and usage possibilities.

While analyzing the works of different authors, it was determined that the following main factors affect the surface roughness the most: species of wood, mode of surface milling, the rounding radius  $\rho$  of the cutting edge, cutting v and feed u speeds (Richter et al. 1995, Buehlmann et al. 2001, Bledzki and Faruk 2005, Ohta and Kawasaki 1995, Touratier 1999, Keturakis and Lisauskas 2010, Keturakis and Juodeikienė 2007, Keturakis and Jakubauskaitė 2009).

Wood is an anisotropic material. The wooden cells that form the anatomical unevenness of the wood surface are cut or deformed during the mechanical processing. When the mechanical processing of the wood is analyzed, the anatomical unevenness of the surface is usually not taken into account, because it is small if compared to the mechanical ones (Volynskyj 2000).

Postulates of the classical wood cutting theory states that the surface roughness increase with the wear of cutting tool. The main cause of the tool wear is considered to be mode of the friction to the wood. The changes in the form of the cutting edge mainly depend on the angular and microgeometrical parameters of the tool (Keturakis and Lisauskas 2010, Zotov and Panfilov 1991, Ivanovskyj et al. 1971).

When the feeding per cutter grows, the kinematic unevenness increases and the quality of the surface gets worse. The waviness of the surface is attributed to the kinematic unevenness. It is

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formed by the rotating movement of the milling tool. The size of the kinematic unevenness depends on the amount of the cutters taking part in the cutting process, cutting radius, feeding per cutter and cutting speed (Keturakis and Juodeikienė 2007, Keturakis and Jakubauskaitė 2009, Ivanovskyj et al. 1971)

According to the results of various tests, when the cutting speed increases, the quality of the surface always improves. For the cylindrical milling of the planes the recommended cutting speed is from 35 to 55 m/s. In this range of the cutting speed the best quality of the surface and the smallest numeric values of the cutting forces are achieved (Ivanovskyj et al. 1971).

The objective of present tests was to determine the influence of the cutting path and rounding radius of the cutting edge on the surface roughness, when the wood samples of pine and black alder are milled along the fiber at different cutting and feeding speeds.

## MATERIAL AND METHODS

The testing samples were made from the wood of pine grown in Lithuania (*Pinus sylvestris*) and black alder (*Alnus glutinosa*) (Table 1). In total 60 samples were prepared for testing, which length was 1000 mm, width was 100 mm, and thickness was 45 mm. The average temperature in the testing room was  $t = 18\pm2^{\circ}$  C, while relative air humidity was  $\varphi = 60\pm5$  %.

<b>Table 1.</b> Physical characteristics of wood.								
Wood species	Moisture content $\omega, \%$	Number of annual rings per 1 cm	Average width of annual ring, mm	Density, kg/m <sup>3</sup>				
Pine (Pinus sylvestris)	10.9	4.21	2.37	504				
Black alder (Alnus glutinosa)	9.44	3.92	2.55	457				

The high-speed steel (HSS 18%) milling knives were used for the tests (Table 2). The chemical composition of the steel (Table 3) was determined by the device used for analysis of X-Ray diffraction (DRON-3). Before the tests, all the knives were sharpened in the same conditions and then the blades were converged.

Fable 2. Specifications of m	illing tool.
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Steel	HS 18 Y28	Dimensions of milling blade, mm	60×30×3
Hardness	67.6 HRC	Sharpness angle $\beta$	40°

Table 3. Chemical com	position of HS	18	Y28 steel.
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Chemical composition of the steel, %										
С	Si	Mn	Р	S	Cr	Мо	Ni	W	V	Fe
1.20	0.20	0.17	0.01	0.02	4.13	0.14	0.09	14.3	3.50	76.3

The tests were done in the stand for wood cutting, which was made in the base of thickness planer (SR3-6). The samples were processed, according to the scheme of the longitudinal milling, when the directions of the cutting speed v and feed speed u vectors are opposite. The conditions of milling test are presented in the Table 4. Two knives were fastened in the cylindrical head of the knives, but only one took part in the cutting process. The second was used for balancing compensation. The thickness of the shaving a, mm was changed indirectly, through the feeding per cutter  $u_z = 0.50$  and 1.00 mm.

The main characteristics describing the wear of milling tool was selected to be the rounding radius  $\rho$ ,  $\mu$ m of the cutting edge. The factual values of rounding radius were determined using the method of lead prints [10] and optical microscope (Nikon Eclipse E200) with digital video

camera (Lumenera Infinity 1). The values of the rounding radius of the cutting edge were determined in the following intervals of cutting path L: 0; 50; 100; 150; 200; 400; 800 and 1600 m. The measurements were done five times in each interval of the path. The received results were processed and measured using the personal computer and software (Infinity Analyze Release 5.0.2). The error of radius measurement accuracy was  $\pm 2 \mu m$ . The received results were processed using the methods of mathematical statistics.

Table 4.	Milling	test	conditions
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Cutting speed v, m/s	22, 41	Width of milling <i>b</i> , mm	45
Feed speed $u$ , m/min	2, 4, 8	Cutting circle diameter D, mm	103
Feeding per cutter $u_z$ , mm	0.50; 1.00	Number of cutting edge z, unit.	1
Depth of milling <i>h</i> , mm	2	Cutting angle $\delta$ , degree	60

The parameter of the processed surface roughness  $R_z$ , µm was measured by contact stylus tip profilometer (MarSurf PS1), the radius of diamond tip of which was 2 µm, measurement angle 90°, and measurement length was 17.5 mm. The surface unevenness was measured in the intervals of cutting path *L*: 50; 100; 150; 200; 400; 800 and 1600 m. Five sectors were selected in one sample (17.5×17.5 mm), and their roughness was measured along and across the fiber. In total 280 measurements were done during the testing series. All the measurement results were processed by Gaussian digital filter. The measurement error of unevenness did not exceed ±10 %.

#### **RESULTS AND DISCUSSION**

The tests helped to determine the influence of the cutting path and rounding radius of the cutting edge on the surface roughness, when the wood samples of pine and black alder are milled along the fiber at different cutting and feed speeds.



**Fig. 1.** Impact of cutting path L on the rounding radius  $\rho$  of the cutting edge: **a** – when v = 22 m/s and  $u_z = 0.50$  mm; **b** - when v = 22 m/s and  $u_z = 1.00$  mm; **c** – when v = 41 m/s and  $u_z = 0.50$  mm; **d** – when v = 41 m/s and  $u_z = 1.00$  mm.

d

While analyzing the received results on tool's wear (Fig. 1), it was analyzed that the tool gets worn the most intensively in the first wear stage until the limit of cutting path 400 m. In the first stage of wear the species of wood does not have any significant influence on the wear

intensiveness. The difference between the values of the rounding radius of the cutting edge in case of milling pine and black alder samples was from 5 to 7 %. In this stage the wear of tool is expressed by mechanical crumbling of cutting edge. When the cutting path reaches the distance of 400 m, the tool's wear gradually passes to the stage of monotonous wear. In this stage the intensiveness of increase of rounding radius is reduced. The tool gets worn evenly, and the microgeometry of cutting edge changes because of temperature and electrochemical corrosion (Zotov and Panfilov 1991). The tool's wear was observed until the section of 1600 m of cutting path.

When the influence of the wood species on tool's wear was analyzed, it was noticed that the tool gets worn more intensively when black alder is milled, although the density of black alder's wood is smaller if compared to the pine wood (Table 1). However, the exception was also noticed: when the feeding per cutter is 0.5 mm, and the cutting speed grows from 22 to 41 m/s, the tool's wear is smaller if compared to the milling results of pine wood. When the cutting speed increases, the bulk part of the shaving loses contact with the processed surface due to the pine's tendency to split under the influence of smaller cutting forces. The shaving is formed more easily; the real length of cutter's contact with the wood gets reduced when compared to the milling of black alder that tends to split less.

While analyzing the influence of feeding per cutter on the rounding radius of the cutting edge, it was determined that when the feeding per cutter grows from 0.5 to 1.0 mm, the intensiveness of the tool's wear gets smaller. When the feeding per cutter is 0.5 mm, the most intensive wear of the tool takes place when the black alder is milled. When the pine wood is milled, the tool gets worn 4 % less if compared to black alder.



**Fig. 2** Surface roughness  $R_z$  along the fiber: **a** – when v = 22 m/s and  $u_z=0.50$  mm; **b** – when v = 22 m/s and  $u_z=1.00$  mm; **c** – when v = 41 m/s and  $u_z=0.50$  mm; **d** – when v = 41 m/s and  $u_z=1.00$  mm.

The received results are interesting for interpretation of the postulates of classical wood cutting theory. With increase of feeding per cutter, the shaving's length gets bigger a little, as well as theoretical contact of cutting edge with wood. The intensiveness of tool wear should grow, but the thickness of shaving also increases, and the tendency of dry wood to split in case of against feed milling results in reduction of real friction contact and wear of cutting edge.

While analyzing the results of surface roughness (Fig. 2 and 3), it was noticed that the surface roughness of pine wood is smaller than that of black alder. This regularity does not change when the surface roughness is measured along and across the wood fiber. The done tests confirm the theory that in case of bigger density of the wood and smaller width of rings

(Table 1), the smaller surface roughness is received. Although the density of pine wood is bigger only by 9 %, but the received surface roughness  $R_z$  along the fiber is smaller on average by 11 %, and across the fiber – by 14 %.

While analyzing the diagrams of the results, it was determined that the increase of the cutting path and rounding radius of tool's cutting edge causes the worsening of the processed surface. This tendency was noticed in all the cases of cutting speed and feeding per cutter. The smallest surface roughness is achieved while milling up to 200 m of the cutting path. From 200 to 800 m limit the surface roughness along and across the fiber increases gradually. The biggest numeric values of the surface roughness  $R_z$  were achieved with cutting path of 1600 m.



**Fig. 3.** Surface roughness  $R_z$  acros the fiber: **a** – when v = 22 m/s and  $u_z=0.50$  mm; **b** – when v = 22 m/s and  $u_z=1.00$  mm; **c** – when v = 41 m/s and  $u_z=0.50$  mm; **d** – when v = 41 m/s and  $u_z=1.00$  mm.

When the feeding per cutter is increased, the surface roughness also increases. The best quality of the surface is received when the feeding per cutter was 0.5 mm, and it is worse at 1.0 mm. This dependency does not depend on the sort of wood and cutting speed. The smallest increase of numeric values of surface roughness  $R_{z}$  was noticed while processing pine wood.

When the cutting speed is increased from 22 to 41 m/s, the surface roughness gets smaller. This regularity is characteristic for both sorts of wood. When the cutting speed is increased from 22 to 41 m/s, the roughness of pine wood is decreased on average by 12 % and that of black alder – by 7 %. The reducing regularity of surface roughness remained at various segments of cutting path when the cutting speed was increased. It is possible to state that with increase of cutting path the wear of the tool gets more intensive. When the wood is milled with blunt tool, the irregular milling process takes place, during which the processed surface is formed when the top layers of the fiber are deformed and compressed.

#### CONCLUSIONS

1. The milling tool gets worn the most intensively when the first 400 m of cutting path are milled. After 400 m of cutting path the milling tool's wear gradually passes to the stage of monotonous wear.

2. The tool gets worn more intensively when black alder is milled. When pine wood is milled, the tool's wear is reduced by 6 %.

3. With increase of feed and cutting speeds, the intensiveness of the wear of milling tool is reduced.

4. The rounding radius of the milling tool's cutting edge has the biggest influence on the roughness of the processed surface along and across the fiber. The best quality of the surface is received when sharp ( $\rho \le 13 \mu m$ ) milling tool is used in the segment up to 400 m of the cutting path. When the rounding radius of the tool's cutting edge is increasing, the quality of the processed surface gets worse.

5. When the feeding per cutter is increasing, the quality of the processed surface gets worse. The best quality of the surface is received when the feeding per cutter is 0.5 mm, and it is a little worse when 1.0 mm. This dependency does not depend on the sort of wood and cutting speed.

6. When the cutting speed increases, the quality of the processed surface gets better. The best quality of the surface is received when the cutting speed is 41 m/s, and it is a little worse when 22 m/s.

7. The quality of the surface of pine wood processed in the same conditions of milling regime is better than that of black alder. The surface roughness  $R_z$  of pine wood along the fiber is smaller on average by 11%, and across the fiber – by 14 %.

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# EFFECT OF KERFING ON CRACK FORMATION IN SCOTS PINE LOG HOUSE TIMBER

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## ABSTRACT

In Norway log buildings are normally produced from logs canted on two sides. The canted faces are prone to crack formation during drying. This can cause some disadvantages, e.g. the cracks can trap water from rainfall, because the canted faces form the wall surfaces in the log buildings. The objective of the reported study was to apply saw kerfs on the upper side of the cants prior to drying to reduce crack formation in the canted faces. The material consisted of 150 mm wide cants produced from Scots pine (*Pinus sylvestris*) logs with a mean top-end diameter under bark of 23 cm. Immediately after canting the cants were divided into four groups: A Chain saw kerf to pith, B Circular saw kerf (9 cm deep), C Circular saw kerf (4.5 cm deep), D Untreated control. The timber was air dried and stored under roof for 4 years. Widths of cracks in the side faces were measured. The results showed that mean crack width was reduced by 60 % by applying chain saw kerf (treatment A). The two other treatments had limited effect on crack formation in the side faces.

Key words: Log building timber, kerfing, crack formation, Scots pine

## INTRODUCTION

Traditional log buildings are usually made of large dimensioned boxed pith timber. In Norway log buildings are normally produced from logs canted on two sides. The canted faces are prone to crack formation during drying. Such localization of the cracks can reduce the quality of wooden structures in several ways. They can reduce the duration of the work are exposed to outdoor climate as it tends to accumulate moisture in the cracks, which causes the environment of decay fungi is favored. The cracks will also result in increased heat loss through walls and thus lead to increased energy consumption in the building's use phase. In addition, cracks appear aesthetically displeasing.

Cracks are caused by mechanical stresses during drying. Drying stresses occur because of the differential shrinkage between the outer part of a log and the interior part. Early in drying, the fibres in the surface dry first and begin to shrink. However, the core has not yet begun to dry and shrink; consequently, the core prevents the outer part from shrinking fully. Thus, the outer part goes into tension and the core into compression. On

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large dimensions the mechanical stresses exceeds the elastic limit of the wood matrix and cracks occur. In addition to the moisture gradient during drying stresses are formed due to the anisotropic shrinkage of wood.

Inducing artificial cracks can be a tool to control the location of cracks to positions where they are less harmful. One possible solution can be to initiate cracks by driving wedges into log building timber (Vreim 1975, Steen 1996, Clementz and Flatland 2008). However, this is a laborious method, and it appears not to have been recurrently practiced.

The objective of the reported study was to evaluate different types of saw kerfs as a rapid method to reduce crack formation during drying of log building timber.

## MATERIAL AND METHODS

In April 2005, 39 pine logs were canted on two sides and debarked to produce 150 mm thick log building timber. All logs had a length of 5 m. The logs were purchased from a harvest of a 125 years old Scots pine (*Pinus sylvestris*) stand in Ringerike municipality performed by Viken Skog BA.

Average top-end diameter under bark was 23 cm, varying from 21 cm to 26 cm (Table 1).

Treatment	No. of logs	Top-end diameter (cm)				
	Ν	Mean	St. dev.	Min	Max	
A: chain saw to pit	10 (5 butt log/5 second log)	22,5	1,2	21	24,5	
B: circular saw 9 cm	$10 (5 \text{ butt } \log/5 \text{ second } \log)$	22,4	1,1	20,5	23,5	
C: circular saw 4,5 cm	$10 \; (5 \; \text{butt } \log/5 \; \text{second } \log)$	22,3	1,2	20,5	23,5	
D: control	9 (4 butt log/5 second log)	22,7	1,2	21,5	25,5	

**Table 1.** Number of logs and top-end diameter for each treatment

The treatments A, B and C (Table 1) were carried out immediately after canting and debarking. One longitudinal saw kerf was produced on the upper side, referring to the position in a wall, of the logs (Fig. 1). The saw kerfs started and ended 50 cm from the log ends. Kerf width for treatment A was about 10 mm, while kerf width for treatments B and C was 3 mm.

The log house timber was air dried and stored under roof until late summer of 2009. Then the timber was stored in a room with 20 °C and 65% RH for about two months before registration of cracks.



Fig. 1. Illustration of the treatments (Illustration: Sigrun Kolstad).

Cracks were recorded on the side faces of the cants with an electronic caliper (Mitotoyo CD-20CX) (Fig. 2). The width of the crack across the length direction was measured at the surface. Measurements were performed 50 cm from each end, and every 50 cm along the cant. Average crack width of a cant was calculated as the average of all the crack width measurements made on both side faces (Figure 2).

Samples for calculation of wood moisture content were extracted 2.5 m from the ends of the cants. The samples were taken from three radial zones: 0-3cm, 3-6 cm and 6-9 cm from cambium at the opposite side of the kerf. The weight of the samples was measured with a Mettler Toledo HB43-S Halogen drying balance

Comparisons of different treatments were carried out by one-way analysis of variance (ANOVA). Tukey-Kramer analysis was used for multiple testing. Effects with a probability of type 1 error smaller than 0.05 were considered significant. The statistical analyses were performed using JMP 8.0 (SAS Institute).



Fig. 2. Illustration of crack detection (Illustration: Sigrun Kolstad).

## **RESULTS AND DISCUSSION**

The prevailing crack pattern was one continuous crack along each side face of the cants.

The results show that the average crack width in the side faces was narrower for all three treatments (A, B and C) compared with the control (D). The effect was not significant for the two treatments with the use of circular saw (B and C). There was also little difference between the circular saw kerf with depth of 4.5 cm and 9 cm. Average crack width was significantly lower in the logs with the chainsaw kerf to pit (treatment A). The average crack width for these cants was 58 % narrower than in the control cants.

Treatment	Ν	Mean	St. dev.*	Min*	Maks*
		(mm)	(mm)	(mm)	(mm)
А	10	1.5	0.81	0	4.3
В	10	2.7	1.78	0.3	8.0
С	10	2.9	1.82	0	9.7
D	9	3.6	1.96	0	7.5

Table 2. Crack width in the side faces of log house timber.

\* Standard deviation, min and max values are based on all measurements within each treatment.

Average crack width was not significantly different in cants from the butt or second logs. This is probably because the logs had about the same top-end diameter, regardless of what part of the trunk they were cut from.

Wood moisture content in different radial zones of the cants is shown in Table 3. There were no significant differences or trends in moisture content between treatments, but the moisture content rose slightly from the surface towards the pit of the cants.

Table 3. Wolsture distribution in the log house timber			
Depth from	Ν	Moisture	Standard deviation
cambium (cm)		(%)	(%)
0-3	18	13,8	0,9
3-6	18	15,4	1,3
6-9	18	16,1	1,8

**Table 3.** Moisture distribution in the log house timber

The Norwegian industry standard for log buildings (Kvalitetskontrollen Norsk Laft 2009) requires that the average moisture content of log building timber is 20 % or lower. The range of individual values shall be within + 3 %/ - 5%. Moisture content should be measured with electrical resistance meter with an insertion depth of 30 mm.

Finstad and Sandland (2009) measured wood moisture content in the walls of two log buildings in Eastern Norway in February, June and October. One was a heated building, and the other a storage house without heating. The measurements were made in 1 cm and 5 cm depth from the inside and outside of the walls. In the heated building, the average wood moisture content at the three times varied between 8.4 % and 11.1 % for measurements made from inside and between 10.9% and 12.9% for the measurements done from the outside. In the storage house, the average wood moisture varied between 13.2% and 16.5% for the measurements from the inside of the wall and between 14.4% and 19.0% from outside.

In the present study the moisture content was slightly higher than that Finstad and Sandland (2009) found in the walls of a heated log building. Although it cannot be excluded that the cracks could develop something more if the timber in this study were used for a heated building there is no reason to assume that this will affect the relative

efficacy of different treatments, since there was very little variation in wood moisture treatments.

## CONCLUSIONS

The results from this study shows that cutting longitudinal saw kerfs in canted log house timber prior to drying can reduce the cracking in the side faces substantially. The largest crack reduction was achieved by using chainsaw with saw cuts to the pit. In these cants the average crack width was almost 60 % narrower than in the untreated control cants. Using a circular saw with a cutting depth of 9 cm or 4.5 cm had a limited effect on the average crack width in the side faces.

Since kerfing with a chainsaw can be performed fast, this is a method that may be appropriate to use if one wants to limit the crack formation in log house timber during the drying process.

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Vreim, H. 1975. Laftehus: tømring og torvtekking. 5.utg. Noregs boklag, Oslo. 74 pp.
# PELLETIZING OF SOFTWOOD AND HARDWOOD WITH SINGLE CHANNEL PELLET PRESSES

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#### ABSTRACT

Pellets made from wood sawdust are popular heating fuel in the Nordic countries. In pellet press, sawdust is compressed into a solid pellet, and it has experimentally been shown that temperature, moisture content and extractives content affect pellet properties. Mechanical durability is an important property since mechanically weak pellets tend to break during storage and transportation. Tree species behave differently in the process, and optimizing energy consumption and pellet quality requires detailed knowledge about the process taking place in the pellet press.

This study aims to characterize those manufacturing parameters that affect mechanical properties of pellets, and to study the behaviour of different tree species in the pelletizing process. We focus on density of pellets and flow conditions in the press channel. Mechanical strength of wood depends on density, and same is probably applicable to pellets. Several tree species are used in pellet manufacturing, and different tree species probably have different flow conditions in the press channel.

We have developed two single channel pellet presses in our laboratory. The first one contains a closed-end die and the second one an open-ended die. The latter one resembles more closely an industrial scale pellet press. Both experimental devices are electrically heated. Temperature and load can be recorded. Pellets were tested with tumbling and compression tests. Durability, strength, and density were reported.

According to our results, density of a pellet increases first rapidly as compression force increases. At a certain load there is a threshold value, after which pellet density only slightly increases when load increases. Silver birch pellets were stronger than Norway spruce or Scots pine pellets at a given load. Mechanical strength of pellets depends on density: increase in density enhances strength. Production temperature also increases pellet strength and durability. In experiments with open-ended die, it was found that there are sharp load drops and oscillating flows in the press channel when sawdust is forced through the channel. Large load drops are problematic, since they may cause excessive wearing of machinery.

Key words: Pelletizing, single channel, softwood, hardwood.

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## INTRODUCTION

Pellets made from wood have become an important fuel in recent decades, and the production, trade and use of pellets has expanded rapidly. Pellets have several advantages compared to other biomass based fuels, such as high calorific value, easy transport and low dry matter losses in prolonged storing. (Heinimö and Junginger 2009)

Main problem with pellet heating in small scale boilers is crumbling of pellets during storage and transport (Ståhl and Wikström 2009). Impacts and friction disintegrate pellets back to sawdust, and the formed fines cause problems with boilers (Ståhl and Wikström 2009). Sufficient mechanical durability should be reached in pellet manufacture, and this is also noted in biofuel standards: European CEN standard for solid biofuels sets limits for durability of pellets (Anon. 2005).

Mechanical properties of pellets, such as strength, depend on raw material properties and process conditions in the press channel. Temperature in pelletizing is an important process parameter: increase in temperature increases the strength of pellets (Rhén et al. 2005, Wild et al. 2007, Nielsen et al. 2009). Laboratory experiments show that increasing moisture content of sawdust decreases pellet strength (Rhén et al. 2005, Nielsen et al. 2009), and also decreases the work needed to compress sawdust through the press channel (Nielsen et al. 2009). Tree species also affects pellet properties: at a given manufacturing temperature, European beech pellets were notably stronger than those of Scots pine (Nielsen et al. 2009).

The compression force in a pellet press is another process parameter, but unlike temperature, its effect on mechanical strength of pellets is somewhat unclear. Rhén et al. (2005) reported that compression force had no effect on the strength of pellets when the pellets were made with a laboratory scale pellet press. On the other hand, Wild et al. (2007) used a similar kind of device in their experiments and found that mechanical strength increased along with increasing compression load.

Pellets are formed by forcing sawdust through a narrow die channel. The length and shape of the die channel have probably a significant effect on the pellet durability, but so far there seems to be lack of studies in this issue. This is understandable, since manufacturing a series of different dies for large rotary presses is expensive. The problem can be overcome by developing a single channel pellet press, in which press channels are comparatively cheap and can easily be changed.

Single channel pellet presses are often used in laboratory scale pellet studies (Rhén et al. 2005, Wild et al. 2007, Nielsen et al. 2009, Stelte et al. 2011). This type of a device has commonly a cylindrical die, which has only one open end. Sawdust is placed into the cylinder, and compressed with a piston against the bottom of the die. The operation of the device, and processes taking place during pelletization, closely resembles an industrial scale press. In this study, we will use a closed-end die to experiment the effect of compression force on pellet properties.

Since sawdust is compressed in the closed-end die against the bottom of the die, the pelletization is not completely similar to the industrial press, where sawdust is forced to an open-ended cylindrical duct. We have also developed a single channel device, which has an open-ended die, similar to dies in industrial scale pellet presses. Continuous

operation of the device can be reached if sawdust is fed automatically. Earlier an openended single channel press has been developed by Nielsen et al. (2009).

The aim of this study is to test the effect of raw material and process conditions on pellet durability with two single channel pellet presses. We will test the effect of compression pressure, die temperature, and tree species on mechanical properties of pellets. In compression pressure tests, we will use Scots pine (*Pinus sylvestris*), Norway spruce (*Picea abies*), and silver birch (*Betula pendula*), and in temperature tests Scots pine and trembling aspen (*Populus tremula*).

## MATERIAL AND METHODS

Two pelleting devices were constructed in our laboratory. The first one consists of a cylindrical die, where one end is closed. Sawdust is placed in the upper end, and a piston then compresses the sawdust into a pellet. The bottom end of the die is then opened, and the pellet is removed from the die. The die is heated with an electrical heating tape. A piston operated by a materials testing machine (Lloyds EZ 50, Lloyds Instruments Ltd., Leicester, UK) compresses sawdust in the upper part of the die channel. The testing machine also recorded compression force for later analyses.

The second single channel press contains an open-ended die, and is also operated by the materials testing machine and heated with the heating tape (Fig. 1). The device comprises a sawdust feeding unit. The die channel is open-ended, and has a wider upper part (13 mm) and a narrower lower part (10 mm). The length of the narrower part was 45 mm. Sawdust used in the tests was sieved for removal of sticks, and conditioned to a moisture content of 10% in a weather chamber (Weiss Umwelttechnik).

Mechanical strength of pellets was tested either in compression or tumbling tests. Compression tests were carried out with the materials testing machine. Tumbling tests were made with a 2000 Geno/Grinder shaker, manufactured by Spex SamplePrep LLC (Metuchen, New Jersey, USA). From each pellet sample a subsample of 7–8 g was conditioned to a moisture content of 10%, weighted and placed into a plastic tube. The Geno/Grinder device shakes tubes in vertical direction, and was set to rotation speed of 1500 rpm for 20 minutes. After shaking, pellets were sieved with a sieve of 4 mm hole diameter, and the mass of particles that did not pass through the sieve holes was weighted and compared to the weight before tumbling. Two softwood pellet samples ( $\emptyset$  8 mm) provided by a commercial vendor, were also tumbled for reference.



Fig. 1. Layout of the device with an open-ended die. Pneumatic actuator feeds sawdust into the upper part of the die.

#### **RESULTS AND DISCUSSION**

Compression pressure had a notable effect on the breaking strength of pellets made in the closed-end die. Fig. 2. shows how strength of pellets increases as pressure increases. After pressure has reached a certain threshold level, there is only a slight increase in the breaking strength. Norway spruce and Scots pine behave quite similarly in the test, but silver birch pellets were significantly stronger than softwood pellets.



Fig. 2. Effect of compression pressure on the breaking strength of pellets. Pellets in these experiments were made with a closed-end die.

In the tests, it was also found that increasing pellet density enhances the breaking strength. This is probably the reason why higher compression forces yield stronger pellets. Highest densities obtained in the tests were about 1300 kg/m<sup>3</sup>. This is close to the cell wall density, which is approximately 1500 kg/m<sup>3</sup> (Kellog and Wangaard 1969).

Temperature experiments were made with the open-ended die. It was found that Scots pine can easily be pelletized in temperatures from 40 to 140 °C, whereas aspen requires substantially higher temperatures from 120 to 180 °C. Microscope images of pellets show nicely the effect of die temperature on the pellets. There are small cracks on the surface of pellets made in lower temperatures. In high temperatures, a solid film forms on the surface of the pellet. It probably gives some additional strength to the pellet.

Tumbling tests also confirm the effect of temperature: increase in manufacturing temperature increases the pellet strength (Fig. 3). Aspen pellets made in low temperatures are generally weaker than pine pellets, but if temperature is 180 °C, aspen pellets become stronger than pine pellets. This probably comes from the chemical composition of hardwood and softwood lignins. We hypothesise that hardwood lignin softens and flows to the surface of the pellet in higher temperature than softwood lignin.



Fig. 3. Effect of the die temperature on the mechanical strength of pellets. Y-axis shows the mass of pellets after tumbling compared to the original mass.



Oscillating load was observed in the compression phase of the open-ended die. When the piston compresses sawdust into the channel, load first rises smoothly and then rapidly as Fig. 4 shows. After the force has reached a maximum value, it suddenly drops as sawdust starts to flow into the channel. The flow is not steady, as can be seen from the figure, and the flow starts to increase again. This kind of oscillation continues few times. Oscillating load is probably due to a phenomenon called stick-slip friction, which is typical for systems where static friction is larger than kinetic friction (Sheng 2008). Oscillating loads may cause vibrations and wearing of machinery.

#### CONCLUSIONS

Increase in compression pressure and die temperature in the pellet manufacture increase mechanical strength of pellets both in compression and tumbling tests. High die temperatures tend to form a film on the surface of the pellets, which also gives them some additional strength. Norway spruce and Scots pine act quite similarly in the closed-end die tests, whereas silver birch with a higher wood density is clearly better regarding pellet strength. Trembling aspen requires much higher temperature in pelletizing than Scots pine, which probably comes from different chemical composition of lignin between softwood and hardwood.

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# MOISTURE CONTENT OF NORWAY SPRUCE STUMP WOOD AFTER HARVESTING

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#### ABSTRACT

In this study the moisture content of Norway spruce stump wood after harvesting was examined. The material was collected immediately after harvesting at the clear cutting area and after different drying times at the roadside storage sites. Immediately after harvesting the average moisture content of stump wood (wet basis) was 53 %. The stump wood dried fairly fast during spring and summer being 31 % one month after stump harvesting. If the stumps dried well a once in the summer, water absorption was fairly slow in the autumn. Each spring and summer the moisture content of the stumps was lower than during the previous year. The moisture content of stump wood followed an upwards opening parabola over a one year period and was repeated each year. Annually the lowest moisture content was observed at the beginning of July and the highest at both the beginning and the end of the year. Three years after harvesting the heating value of the stump wood at the storage was still 5.2 MWh/ton. On the whole, the stumps at the forest roadside storage sites were combustible at any point during the three year period except a one month drying period immediately after harvesting. The original research article: "Laurila, J. & Lauhanen, R. 2010. Moisture Content of Norway Spruce Stump Wood at Clear Cutting Areas and Roadside Storage Sites" is in print (Silva Fennica a Journal of Forest Science vol. 44(3), 2010).

Key words: bioenergy, harvesting, moisture content, Norway spruce, stump wood.

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## INTRODUCTION

The role of renewable energy is an essential part in improving energy self-sufficiency and preventing climate change. In 2008 the final energy consumption of renewable energy in Finland was 30.5 % of the total energy usage. The aim of the European Union Commission is to increase renewable energy usage in Finland to 38 % by the year 2020 (Energy.eu. 2010). The target is a very challenging one for today's energy production technology. Therefore all potential and cost effective bioenergy sources and new innovations are needed.

The harvesting of Norway spruce (*Picea abies* (L.) Karst.) stump wood has increased recently in Finland where stump wood is an important source of bioenergy. Crushed stump wood is a suitable biofuel for large power plants where small impurities do not cause problems (Backlund 2009, Hakkila and Aarniala 2004). Stump wood has a high energy content. The average energy content of stumps is about 130 MWh/ha and can even be as high as 250 MWh/ha (Hakkila 2004, Näslund Eriksson and Gustavsson 2008). However, cost effective energy generation from stump wood depends on strongly its moisture content.

Norway spruce is loosely anchored in the forest soil and therefore spruce stumps are easier to harvest than pine stumps (Hakkila 1972, Laitila et al. 2008). Stump harvesting has also some positive effects on forest regeneration. For example it replaces traditional soil preparation when reforesting and can result in a reduction of the infection rate of root rot fungus. Also, the damage of pine weevils might have reduced. Therefore stump harvesting decreases the costs of reforestation (Procurement of forest... 2003, Saarinen 2006).

The first aim of this study was to clarify the moisture content of Norway spruce stump wood at the clear cutting areas immediately after stump harvesting and at the forest roadside storage sites after different drying times. The second aim was to clarify the correlation between moisture content and other factors such as drying time and air humidity. The third aim was to examine the heating value of stump wood after three years of storage.

#### MATERIAL AND METHODS

The material was collected from four stump harvesting sites in western Finland between June 2006 and May 2009. The stumps were harvested in June - July 2006 and the sites were clear cut one to six months prior to the stump harvesting. The stumps were harvested with excavators. Some weeks after the harvesting, the stumps were moved from the clear cut areas to the forest roadside storages by a forwarder.

The samples for the moisture content analysis were collected from the piles at clear cut areas and also from the forest roadside storage sites randomly. From each of the four stump harvesting sites, the first samples were taken from the clear cutting areas immediately after stump harvesting. The other samples were collected from either clear cutting areas or roadside storage sites after different drying times. Every sample was taken from the surface layer of the piles. The sampling point (Fig 1.) was midway between the stump and the end of the root.



Fig 1. The sampling point.

The moisture content analysis was made according to the international Standard ISO 589 "Hard coal – Determination of total moisture". The drying temperature was 105°C, and a drying time of 24 h was used. Heating value analysis was based on standard: CEN/TS 14918:2005 Solid Biofuels – Method for the determination of calorific value. The weather data was received from Finnish Meteorological Institute and the Finland's Environmental Administration.

#### RESULTS

#### Moisture content and heating value

The average moisture content of the stump wood was 53 % immediately after harvesting with a standard deviation of 6 % (Fig. 2). After stump harvesting the moisture content was getting lower quite quickly (Fig. 3), and about one month after harvesting the average moisture content was 31 %. The lowest recorded moisture was only 13 %. During the autumn of 2006 the moisture content increased and decreased again during the spring of 2007. The same phenomenon was repeated each year during this study. The moisture content was at its highest at the beginning and the end of each study year. Lowest moisture content on average was in the beginning of July. The heating value of stump wood was 5.24 MWh/ton and ash content 1.7 % three years after harvesting.



Fig 2. Moisture content of stump wood immediately after harvesting at four sites.



Fig. 3. Average moisture content of stump wood and air humidity as a function of time.

#### Correlations

There was a weak ( $R^2=0.31$ ) non-linear correlation between stump wood moisture content and air humidity based on the four year average excluding the first moisture content results immediately after harvesting in 2006. Also, there was a non-linear correlation ( $R^2=0.44$ ) between the stump moisture content and the temperature. Moreover, between the moisture content of the stumps and time a non-linear correlation ( $R^2=0.51$ ) was observed, if the first results of the moisture content immediately after harvesting in 2006 were ignored. The highest coefficient of determination ( $R^2=0.63$ ) in this study was received using a four variable moisture content model (Eq. 1).

$$MC = 0.0189t_{wn}^2 - 1.0694t_{wn} - 0.0021AH_a^2 + 0.2499AH_a + 0.0375T_a^2 - 0.7260T_a - 0.0340t_d + 32.747$$
(1)

where:

MC	=	moisture content (wet basis)
t <sub>wn</sub>	=	calendar week number
AH <sub>a</sub>	=	air humidity (weekly average)
Ta	=	temperature (weekly average)
t <sub>d</sub>	=	drying time in weeks

#### DISCUSSION

Moisture content of stump wood is one of the most important factors in the cost effective heat energy production. It directly affects, among others, the cost of transportation and the heating value of the stump wood. The most favourable point in time for stump harvesting is in the spring and early summer. Immediately after harvesting the moisture content decreased rapidly. During the late autumn the moisture content increases again, but not significantly. This might be due to pit aspiration. Every spring and summer the moisture content was at a lower level compared to the previous year. Overall the stumps were combustible at any point during the three year storage period excluding the one month drying period immediately after harvesting.

The moisture content samples were collected from the surface layers of the piles at the clear cutting areas and at the forest roadside storage sites. It was possible that the lower layers of the piles had different moisture content than the surface layer. The variation might be either positive or negative depending on, among others, the weather conditions and the season.

There was almost no deterioration of the quality of the stump wood during the three year storage period. The energy content of the dry mass of stump wood was almost the same after the three year storage period than immediately after harvesting. The ash content of stump wood was lower than usually. Normally stumps include soil remnants which increase ash content. Probably rain and frost cleaned the stumps at the storage during the long storage period.

The results of this study can be used as background information for the calculation of cost-effective stump wood procurement and also for production planning and utilization of stump wood.

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## MECHANICAL PROPERTIES OF FOILED BIRCH PLYWOOD WITH SPECIAL VENEER LAY-UP SCHEMES

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## ABSTRACT

Plywood is a wood based panel material laminated of veneers with grain direction perpendicular in adjacent layers. The usage of plywood can be expanded in cause of possibility to use special lay-up schemes tending to improve mechanical properties which depend on grain direction in outer plies. Also the presence of phenol film and thickness reducing of outer ply may influence plywood strength properties. That's why the research purpose was to determinate the bending strength and modulus of elasticity of foiled birch plywood types with special veneer lay-up schemes, bending flatwise and edgewise. Two special plywood types produced at JSC "Latvijas Finieris", 28 mm thick Spec1 and 30 mm thick Spec3, with different veneer lay-up schemes were chosen for bending properties determination tests according to the European standard EN 789. Specimens were tested with face veneer direction parallel and perpendicular to longitude. As well moisture content and density of a specimen were determined. Report contains bending strength properties comparison of special plywood specimen with different width and load direction. The results are bending strength is significantly higher for less wide special plywood specimens. And the bending strength of special plywood loaded edgewise are 4 % lower for *Spec1* type and 9.5 % lower for *Spec3* type than loaded flatwise for a specimen with same cross-sectional dimensions.

Key words: foiled plywood, veneer lay-up schemes, bending strength, modulus of elasticity.

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# EFFECT OF WOOD CONSTITUENTS OXIDATION ON UNSATURATED FATTY ACIDS

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#### ABSTRACT

The overall aim of this project was to develop the concept of using new vegetable oil based treatments on exterior wood to improve the outdoor durability. The drying mechanisms of the unsaturated fatty acids with wood model system in the real time were monitored by using RT-IR. This method together with solid-NMR is enormously powerful spectroscopy techniques to determine the physical and chemical properties of fatty acids-wood during the oxidation process over time. In the first part of this study the focus was in a molecular level on oxidation of methyl linoleate mixed with lignin model compounds. The effect of lignin structures on unsaturation fatty acids oxidation was determined with 1 wt% lignin model compounds by using RT-IR. The wood model compounds minimize the complexity of all elements that exist normally in the natural wood. In general phenolic lignin or conjugated lignin structures were observed to inhibit the oil oxidation (drying) process. The oil oxidation interaction mechanisms were also evaluated by using methyl linoleate, methyl oleate and Linola® on solid wood. This study indicated that the oxidation pattern of fatty acids behaved differently on wood surface. The results from these studies clearly show that an improved performance of vegetable oil treatments on wood can be obtained by an appropriate choice of oil structure. The general conclusion is that a high enough reactivity to obtain an immobilized oil layer is needed but that a too reactive oil system will reduce the long term durability.

Key words: Methyl linoleate, fatty acids auto-oxidation, lignin, RT-IR, solid NMR

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## STRUCTURAL ANALYSIS OF WOOD BASED CERAMICS

Lautner, S.<sup>1</sup> and Fromm, J.<sup>2</sup>

## ABSTRACT

Wood is a natural tissue complex consisting of various cell types such as tracheids, fibres, vessels and parenchyma cells. It is mainly composed of cell walls with cellulose, hemicellulose and lignin as the major biopolymeric constituents. Recent activities are directed to the question how to transform the hierarchical cellular structure of wood into inorganic materials like ceramics with specific functional properties. With regard to SiC-ceramics derived from wood it was shown that the cellular structure can be transformed into biocarbon templates and SiSiC-ceramics. In order to gain further insights into the structure of these ceramics we analyzed SiC-ceramics derived from wood and wooden composites by light microscopy, SEM as well as by SRµCT at the HASYLAB of the Deutsches Elektronensynchrotron (DESY) in Hamburg. Results showed different porosities of the various materials depending on the infiltration technique; while Si-gas infiltration produced a SiC ceramic with a high porosity liquid Si infiltration resulted in a SiSiC-ceramic with a very low porosity.

Key words: Wood composites, wood ceramics, synchrotron radiation, SiC, SiSiC

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# QUANTIFYING FUNGI IN LOGGING RESIDUES WITH REAL-TIME PCR

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## ABSTRACT

Logging residues, branches and treetops after logging, were considered in the past as unsalable portions of the felled trees and remained on the landing. Currently, logging residues are harvested, stored in piles for variable time periods prior to being utilized as a bioenergy source. However, it is still unclear to what extent the colonization by decay fungi during outdoor storage impairs the fuel quality. Our objective was to find out whether the storage method influenced the amount of basidiomycetous fungi, the main wood degraders in logging residues.

We used fungal DNA quantification with real-time PCR as a novel approach in this field and related the amount of fungi to physical parameters in logging residues measured during the storage. We found that fungal DNA decreased with decreasing moisture in the logging residue over time, but increased with precipitation. Fungal colonization was higher in samples harvested in the spring than harvested during autumn. We also found more fungal DNA in *Pinus sylvestris* than in *Picea abies* logging residues and loose material had higher fungal colonization than bundles.

In conclusion, we show that quantification of fungal DNA is feasible in logging residues, providing accurate information not readily obtainable otherwise. The level of colonization of decay fungi detected using this sensitive method was minor, indicating low level of decay in the logging residues. Our findings indicate that the storage methods or duration of maximum 460 days did not significantly change the fuel quality of logging residues.

Key words: Basidiomycetes, fungal colonisation, forest slash, harvesting residues, wood decay.

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# WOODEN ROOF SHINGLES: IMPROVEMENT OF DURABILITY STRATEGIES

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## ABSTRACT

Wooden roof shingles were made from pine (*Pinus sylvestris* L.) and spruce (*Picea abies* (L.) Karst.). The natural durability of those wood species is classified as class 4 (non-durable). To improve the durability of roof shingles against basidiomycetes, they were impregnated or hydrothermally modified. Impregnation was done with one of two commercially available preservatives - Celcure AC 500 or Dikants. Celcure AC 500 is a copper based preservative, whereas Dikants is chrome-fluorine-boron based. Shingle impregnation with Celcure AC 500 was done in two ways - by dipping and vacuum treatment; impregnation with Dikants was done by dipping. After impregnation, all roof shingles were coated with linseed oil. Hydrothermal modification was done in two regimes -  $150^{\circ}$ C/3 h and  $160^{\circ}$ C/1 h. To determine the efficiency, impregnated or thermally modified roof shingles were tested according to the standards EN 113 (*G. trabeum*) and EN 84 (*C. puteana, G. trabeum* and *P. placenta*).

The first durability class was reached almost for all preservation strategies, except the case of impregnation with Celcure AC 500 by dipping for spruce, where the mass loss was 5.1%. The best results according to EN 113 were obtained for spruce, hydrothermally modified at  $150^{\circ}$ C/3 h and the Celcure AC 500 vacuum treated pine (amount of the chemical preservative  $3.3 \text{ kg/m}^3$ ) - both showed a mass loss of 1%. The results of the tests according to EN 84 indicated that none of the chemical preservatives after leaching was effective against *C. puteana*. Heat treatment was more effective than chemical treatment, and pine gave better results than spruce. For pine, the first durability class was reached in both the hydrothermal modification regimes, but spruce reached the first durability class when it was modified at  $160^{\circ}$ C/1 h.

Key words: decay resistance, heat treatment, Celcure AC 500, Dikants.

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# COMPRESSION PROPERTIES OF THREE LAYER CELLULAR WOOD PANELS

# Iejavs, J.<sup>1</sup>, Spulle, U. & Jakovlevs, V.

#### 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. The aim of the research is to evaluate the compression properties perpendicular to the plane of the three layer cellular wood panels for structural application. There were 8 specimens manufactured with panel thickness 136 or 152 mm, width and length 300 mm of each of the six horizontal load bearing panel structural models. Scots pine (Pinus sylvestris L.) cellular wood material and solid pine wood ribs were used as internal layer of the structural panels. Cellular wood core was placed in horizontal or vertical direction. Scots pine solid wood panels and birch plywood were used as top layer material. Applied glue was polivinilacetate Cascol 3353. Common stress type in structural subfloor panels is compression therefore the influence of the cellular material orientation, ribs and top layer material on the sandwich type structural panel compression strength was evaluated according to LVS EN 408:2011. Extra parameters like moisture content and apparent density were determined. The initial research shows that different structural models have a great effect on the cellular wood material panel compression strength. Cellular wood panels with vertical direction of cellular wood material show significantly higher compression strength compared with panels with horizontal direction of cellular wood material. Wooden ribs significantly increases compression strength of three layer cellular wood panels. This initial study carried out within project "Elaboration of innovative self supporting panels and building elements made of cellular wood material" and results will be used for further development of structural panels of cellular wood material.

Key words: light weight panels; cellular wood; compression strength.

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# INFLUENCE OF ASH WOOD SURFACE ROUGHNESS ON THE ADHESION OF ACRYLIC POLYURETHANE COATING

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### ABSTRACT

For the research the samples of dried (moisture content was 12 %) ash (*Fraxinus* excelsior L.) wood were used. Before wood finishing, the surfaces of the samples were sanded. In order to get different surface roughness the abrasive material of P80, P120, P150, P180, P220 and P240 grit was used. The parameters of surface roughness  $R_a$ ,  $R_z$  and  $R_{max}$  were measured in three directions: along the wood grain, across the grain and in the angle of 45°. The non-linear dependency of roughness parameters was noticed. For evaluation of adhesion strength, the polyurethane coating of clear varnish was obtained. Dimensions of finished samples were  $50 \times 50 \times 15$  mm. Twenty samples were prepared to test each group of roughness. The adhesion strength of wood coatings was assessed using the pull-off method (according to ISO 4624:2002 standard). The obtained results and dependencies are presented in the article.

Key words: ash wood, surface roughness, polyurethane coating, adhesion strength, pull-off testing.

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# METHODS USED TO EXAMINE THE MOISTURE BEHAVIOUR OF THE WOODEN FLOOR OF VIIKKI CHURCH

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## ABSTRACT

Viikki Church, built in 2005 and designed by architect Samuli Miettinen, represents modern Finnish wood architecture. The church has a large wooden floor which has unusual characteristics. Very few large wood floors have been built with the combination of under-floor heating together with a reinforced concrete foundation. Therefore it is not known how the floorboards will behave when subjected to the combination of under-floor heating and natural humidity variation occurring over the year. The floor is made of feathered spruce floor boards (30 mm x 120 mm) sawn between the tangential and radial directions. The floor was treated with a mixture of lye and oil.

This poster presents the methods used in a study about the moisture behavior of the wooden church floor. The study presented is a follow-up to an original study carried out between 2005 and 2006, right after the completion of the church. The aim of the original study was to observe the moisture behavior of the wooden church floor over a period of one year. The follow-up study repeats the same measurements. The aim is to observe the behavior of the floor after it has been in service for 5 years. Of particular importance are the gaps between the floorboards; if they are wide enough to allow moisture movement. Also the moisture content is recorded at different points in the church building.

The humidity of the church hall floor boards were measured with a capacitive humidity meter. Floor deformation, cracks and widths of the floor edges were measured with a measuring tape. The indoor air relative humidity and temperature were recorded constantly. The results of the original study showed that the variation in moisture content was between 6 and 12 %. The gaps between the floorboards were 1.5 to 3.5 mm wide during the dry winter period. Also some recommendations about the maintenance of the floor could be made. The follow-up study is ongoing research, the results will show if the floor behaves differently after five years in service.

Key words: wood, flooring, moisture behaviour, Viikki church.

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# WOOD COMPONENTS AT THE NANOSCALE: ATOMIC FORCE MICROSCOPY AND COMPUTER MODELING

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### ABSTRACT

In wood, cellulose is the main reinforcing component, exhibiting extraordinary mechanical properties when considering its low weight. The unique features of cellulose in combination with its renewable character make it highly interesting for use in the design of new materials and applications. As new processes emerge that allows for separation of wood fibers into nanoscopic components a need to investigate nanomechanical properties and molecular interactions arise. Atomic Force Microscopy in combination with Molecular Dynamics (MD) simulations is a powerful approach for studying interaction forces at the molecular scale. AFM force measurements and MD simulations may be used to study the observed differences in mechanical properties of single cellulose fibrils obtained from various sources, differing only in their fine structure. Additionally, structural variations along a single fibril can be identified under optimum conditions, thus yielding important insight into the distribution of crystalline and less ordered domains of cellulose. Moreover, the manipulation of single polysaccharides by stretching and pulling is possible using AFM single molecular force spectroscopy (SMFS). Such experiments in combination with molecular dynamics (MD) simulations can reveal important mechanical and structural properties of individual polysaccharides or elucidate on their binding mechanisms to other wood constituents. For example the approach can shed further light on the nature of the strong interaction between xyloglucans and cellulose or elucidate on properties implicated for nanocomposite design. Computer simulations are helpful when studying the atomistic interactions that are decisive for the mechanical properties of single cellulose nanofibrils but also for revealing important aspects of cellulose swelling and dissolution properties.

Key words: Cellulose Nanofibrils, Molecular Dynamics Simulations, Atomic Force Microscopy, Single Molecular Force Spectroscopy, Nanomechanics.

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# WOOD PRODUCTS IN A LOW CURRENT PULSING ELECTRIC FIELD – A NEW WAY TO PROTECT WOOD?

# Treu, A.<sup>1</sup> & Larnøy, E.<sup>2</sup>

#### ABSTRACT

A new protection system has been tested which protects wood without treating it - by installing a low pulsing electric field. This electro-osmotic pulsing technology on wood, called PLEOT, has been tested in lab trials.

Wood has a low specific conductivity and is considered as a dielectric material. Water plays therefore an important role. With increasing wood moisture content, a favorable environment for fungi development is created. At the same time, increasing wood moisture content increases the conductivity in wood and PLEOT can protect the material. Wood can be considered as naturally protected against fungal attack at a wood moisture content <20 %. It could be shown in lab tests, that a protection by means of PLEOT can be achieved at higher wood moisture content.

The current study evaluates the potential of this technology as a wood protection system. Different fungi and wood species are tested. The results show that PLEOT fully protects Scots pine sapwood and beech wood samples during 8 weeks when exposed to *Coniophora puteana* and *Trametes versicolor* in laboratory trials. Fungal degradation of untreated wood samples after 4 weeks of colonization could be stopped or slowed down in case of white rot exposure. Wood moisture content after basidiomycete test is lower for PLEOT-protected wood samples than for untreated samples. However, it is assumed that the wood moisture content of PLEOT-protected samples is not below an unfavorable amount for fungal attack.

Further studies will also include other wood-based materials and focus not only on protection against fungal attack by white rot and brown rot, but also include surface fungi and investigate the wood-water relations.

Key words: Electro osmotic pulsing, PLEOT, Wood protection

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