OUTDOOR LOAD-BEARING TIMBER STRUCTURES

Project summary report

ERIK SERRANO (Editor)

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Preface

This report contains an overview of the activities and a compilation of the main results from the project *Outdoor Load-bearing Timber Structures.* The project partners have provided the information and this information has in turn been compiled by me.

The project has been a part of a coordination project ("IPOS – Svenskt trä – Innovationspotential för det biobaserade samhället" [Swedish wood – Potential for Innovation for the Bio-based Society]). IPOS, in turn, is part of *BioInnovation,* a strategic innovation programme funded by the innovation agency Vinnova, by the Swedish Energy Agency and by Formas. The project was also partly financed by Formas grant number 2016-01138 and by The Swedish Federation of Wood and Furniture Industry – Trä- och Möbelföretagen (TMF). The financial support from all these organizations is hereby gratefully acknowledged.

I would like to express my sincere thanks to all project partners for their respective contributions during the project. It has been a privilege to get access to your combined knowledge and experience. Thanks also for your respective contributions in terms of materials and products being sent across Europe, for your time and for the interesting discussions we had. Finally, I would like to thank you for showing outstanding hospitality and openness in general and in particular in connection to the combined project meetings/study visits we had during the last three years.

It has been a true pleasure coordinating this!

Lund, November 2020,

Erik Serrano
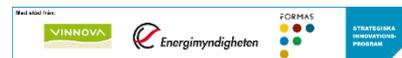
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1   Introduction

1.1   Background

The Swedish stock of multi-storey timber buildings has in recent years increased significantly although the use of wood in outdoor load-bearing applications has been very modest. For outdoor load bearing applications properties like *e.g.* durability, dimensional stability, appearance, strength, stiffness, paintability and glueability need to be taken into account. In many cases domestic (Swedish) wood species without treatment cannot meet these requirements.

Treatment of wood with traditional preservatives helps solving some of the problems (durability), although other of the abovementioned desired features are not met. Furthermore, in many European countries there is pressure against the use of traditional wood preservatives due to their leaching into water and difficulties with their removal at the end of their life. One way to overcome these issues is to use non-biocidal wood modification methods offering the potential to tailor the properties of the timber products to meet the requirements.

Wood modification can be defined as the action of a chemical, biological or physical agent upon a material, resulting in a property enhancement during the service life, excluding traditional preservative treatment with biocides. Several wood modification methods, *e.g.* thermal treatment, acetylation and furfurylation, have been introduced on the Scandinavian building material markets.

Such biocide-free modified wood has a well-documented and significantly improved durability as well as increased dimensional stability compared with unmodified wood. However, their major use has been limited to non-structural applications, and thus there is a need for further research into the topic of load bearing structures.

In this project, acetylated Scandinavian wood species were studied for different types of load bearing applications, including species like pine and birch and including both solid timber and veneers.

1.2   Aims and objectives

The overall objective has been to contribute to an increased added value for the domestic biomass. The project aimed at investigating possible new wood products for use in outdoor load-bearing structures. The development of appropriate connections for such applications is of paramount importance and, consequently, joints have also been studied. Promoting the use of locally produced raw material translates into a sustainable production of biomass enabling in turn am increased use within the construction sector, thus reducing both climate effects and use of fossil-based materials. An increased use of wood in outdoor load bearing structures increases the possibilities for the wood-based industry to compete with other materials. If new materials and components are to be introduced, it is of utmost importance to also study the possible adaption of existing design rules, or if necessary introduction of completely new design provisions. Consequently, the current design provisions of Eurocode 5, have been revisited for a few design situations related to the use of dowel-type joints.

1.3   Project overview

The project has been organised in four work packages, WPs, covering;

- WP0: Administration and project management
    - Organisation of meetings, coordination, reporting to funding organisation
- WP1: Process, modification and wood species (material)
    - Material logistics, acetylation in industrial scale facility, specimen preparation, durability testing.
- WP2: Materials, gluing and surface treatments
    - Gluing of materials, surface characterisation, surface treatments, testing
- WP3: Materials, components and construction (structural design)
    - Manufacturing of specimens, design of test set-ups, mechanical testing (materials and structural components and joints), numerical modelling,
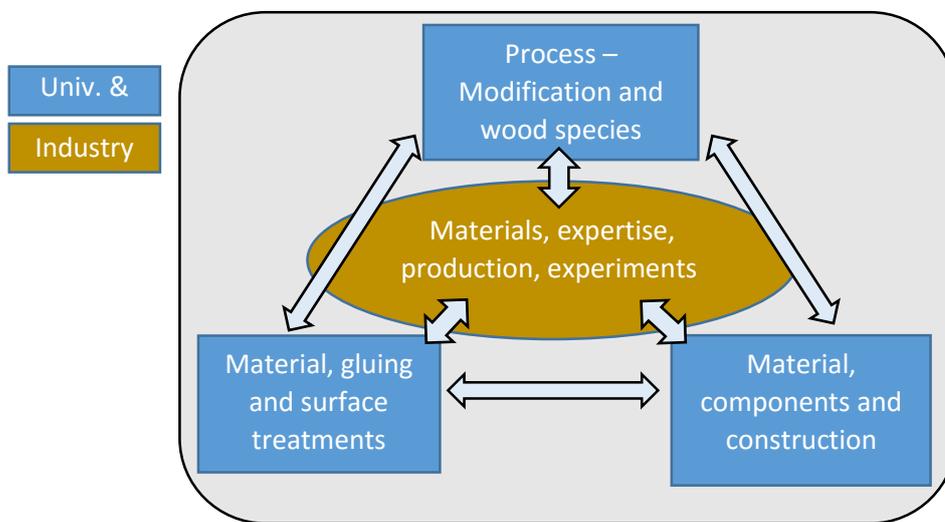
Figure 1. Work packages, academic and industry partner interaction.

1.4   Participants

Academic partners *Lund University, Structural mechanics* *Lund University, Structural engineering* *RISE Bioeconomy* *KTH Building materials*	
Industry associations *TMF*	***Acetylation technology providers*** *Accsys PLC*
Structural design and component development *Novana AB* *Ingenieurbüro Miebach*	***Wood material providers*** *Accsys PLC* *Vanhälls Såg AB* *Koskisen OY*
Fasteners and adhesives expertise *Rothoblaas* *Akzo Nobel AB*	***Component and application expertise*** *Moelven Töreboda AB* *Simonin SAS* *Innoventum AB* *Modvion AB*

1.5 Project Overview – Main tasks, reporting and meetings

The practical work in the project has mainly been organised in a number of tasks relating to material supply, test set-up and component design, acetylation and specimen manufacturing and, finally, characterisation (testing).

The following main tasks have been completed:

1. Material selection and acetylation
    a. Selection of solid pine and solid birch
    b. Manufacturing of Birch veneers for acetylation
    c. Acetylation of solid pine and solid birch
    d. Acetylation of Birch veneers
2. Adhesive system verification, surface characterisation
    a. Gluing of different adhesives
    b. Surface characterisation
3. Component manufacturing
    a. Gluing of Birch Plywood (acetylated and unmodified)
    b. Manufacturing of glued-in rods specimens (Birch)
    c. Manufacturing of glulam beams, including finger jointing
4. Development of test methods for mechanical testing
    a. Design and manufacturing of test rig for beam testing
    b. Adaptation of test method for fracture energy testing
    c. Numerical modelling supporting development of test methods
5. Testing and evaluation
    a. Testing of hybrid glulam beams
    b. Determination of pH and acetyl content (solid wood, veneers, Birch and Pine)
    c. Evaluation of durability
    d. Characterisation of acetylated and unmodified pine and birch: strength, stiffness and fracture energy
    e. Influence of moisture content on mechanical properties of pine and birch (acetylated and unmodified)
    f. Embedment strength of pine (acetylated and unmodified, parallel and perp. grain)
    g. Single dowel joint tests, parallel and perpendicular to grain, (acetylated and unmodified pine).
6. Reporting and communication (see "Research output")
    a. Peer reviewed journal articles
    b. Conference contributions
    c. Master's theses
7. Meetings and interaction within the project.

In total five physical project meetings were held ca every 6 months, at partners' facilities starting in autumn 2017 at RISE Borås (SE). Meetings during 2018 were held at Rothoblaas (IT) and at Koskisen (FI). In 2019 the meetings were held at Accsys (NL) and at Simonin (F)). Due to the pandemic, no further physical meetings were held, but instead meetings in spring and autumn 2020 were arranged as virtual meetings in Zoom.

2   Materials and acetylation

2.1   Acetylation

Acetylation of wood to enhance its resistance against wood decaying fungi and insects, as well as improving its dimensional stability under varying moisture conditions, has been studied extensively over the last decades (Alexander *et al*. 2014, Bongers *et al*. 2015, Goldstein *et al.* 1961, Hill *et al*. 2006, Rowell and Dickerson 2014). Generally good agreement amongst researchers exists in that, at least above acetyl weight percent gains (WPG) of 15-20%, acetylated wood shows marked resistance to attack by most wood destroying fungi (Alexander *et al.* 2014, Beckers *et al.* 1994, Peterson and Thomas 1978, Suttie *et al*. 1999). Data from long running in-ground stake studies (18 years) have confirmed that long term durability against fungal decay is only likely to be achieved at WPG of 22% or above (Larsson-Brelid and Westin 2010).

Accsys Group introduced acetylated wood, named Accoya® wood (www.accoya.com), into the market in 2007. Accoya® wood is based on the acetylation of radiata pine (*Pinus radiata* D. Don) and is mainly used for non-structural applications such as joinery, cladding, decking and (light) civil works (Bongers *et al*. 2009).

The reaction of acetic anhydride with wood results in esterification of the accessible hydroxyl groups in the cell wall to acetyl groups. With increasing level of acetylation directly the amount of moisture that can be bound to the cell wall is reduced. The degree of acetylation can be reliable quantitative determined by a number of chemical analysis methods such as High Performance Liquid Chromatography (HPLC) and Gas Chromatography (GC) and spectroscopic methods like FTIR and NIR (Beckers *et al*. 2003, Schwanninger *et al*. 2011).

2.2   Acetylation of scots Pine, solid Birch and Birch veneers

Acetylation was done by partner Accsys, at the plant in Arnhem, The Netherlands. The Birch material was provided from project partners Vanhälls Såg AB and Koskisen OY (veneers). The scots Pine was delivered by Pölkky OY (not a formal project partner).

2.2.1   Scots pine

Previous research with scots pine (*Pinus sylvestris*) showed consistent acetylation of sapwood (e.g. batch 5228). The acetylation of scots pine heartwood is much more variable and insufficient consistent to make a guaranteed acetylated product.

As part of the Bio-Innovation project "young" Scots pine (32 x 125 mm) was purchased has been X-ray scanned prior to cutting. It is questioned if this heartwood (that is not different in wood moisture content compared to the sapwood) can be consistent acetylated.

Batch 7890
In August 2019 "young" Scots pine (192 boards of 32x125 mm, 4.5 m long) was obtained from Pölkky OY (Finland). Most boards contain pith and heartwood, and some boards were highly resinous (see pictures below). The young Scots pine has been acetylated in batch 7890 according a standard Accoya 25 mm process.

<div align="center">Heartwood (reddish) and pith in Scots pine   High resin content</div>

Figure 2. Examples of raw material used for the acetylation in batch 7890.

Batches 8495 and 8974
In January 2020, 396 boards of X-ray scanned Scots pine were obtained from Pölkky OY (Finland). All boards were numbers such that the X-ray scan could be linked to success of acetylation. Most boards contained pith and heartwood, and a few boards were highly resinous.

In batch 8495 (32 mm radiata pine process) 51 boards were selected based on discussion with the wood supplier on basis of the X-ray measurements. The results were discussed with the company doing the scanning (Finnos OY), and based on the results a prediction model was set up.

To verify the model, 140 boards, of which most boards were predicted as well treatable, were acetylated in batch 8974.

2.2.2   Birch

Circa 1.4 m³ of Birch of 38 mm thickness, random width and 1.5 m long was obtained. The birch was acetylated according to a standard Accsys' radiata pine process (batch 6634).

2.2.3   Birch veneer

As part of the Bio-Innovation project 3 mm thick rotary cut birch veneer was obtained (circa 1.2 x 1.2 m) from Koskisen (Finland). The veneers were acetylated in different batch 6877, 7504, and 7949.

Figure 3. Birch veneers used for acetylation.

2.3 Results and Discussion

2.3.1 Scots pine

Batch 7890

In the table below the results of the acetylated Scots pine are given. From the table it is clear that 40% of the heartwood (4 out of 10) has a too low acetyl content, high acetic acid content and is showing checking / burning spots (see Figure 5). No relation is seen with wood density (see Figure 6).

Table 1. Results from acetylation of Scots pine.

Board	Oven-dry density	Sapwood		Heartwood		Remarks	Heartwood fraction
	[kg/m³]	Acetyl [%]	Acetic acid [%]	Acetyl [%]	Acetic acid [%]		
1	440	√	0,2	√	0,4	Burning spot	30%
2	443	√	0,3	√	0,3	-	30%
3	438	√	0,3	X	1,4	Burning spot and check	20%
4	419	√	0,3	√	0,4	-	30%
5	438	√	0,3	√	0,5	-	30%
6	459	√	0,2	=	0,9	Burning spot and check	20%
7	421	√	0,3	√	0,2	-	30%
8	551	√	0,3	X	1,3	Burning spot and check	30%
9	454	√	0,3	√	0,3	-	50%
10	439	√	0,3	X	1,8	Burning spot and check	20%

* acetyl content good (√), around specification (=), not sufficient (X)
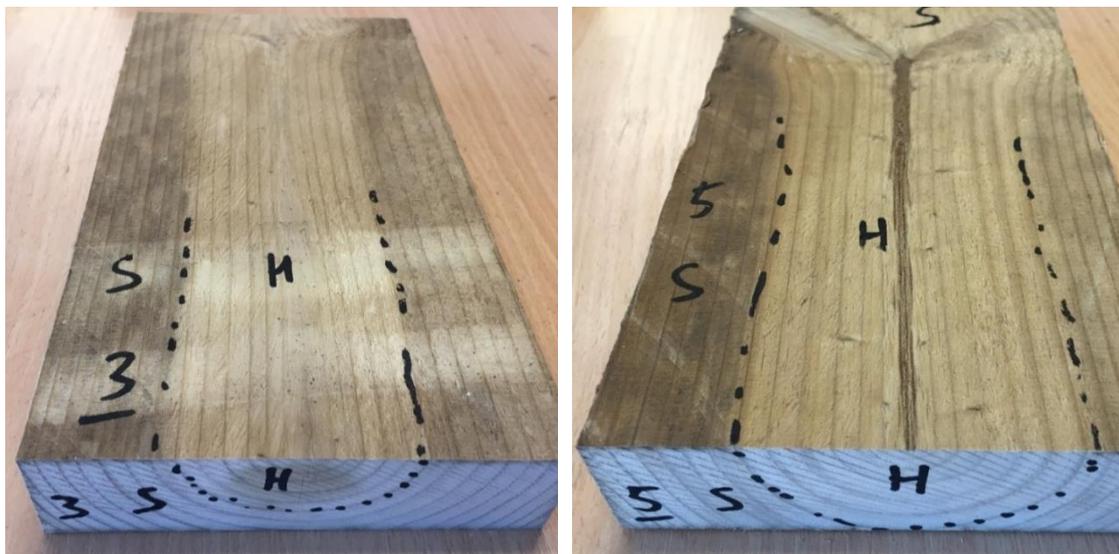
Acetylated Scots pine with heartwood (H), sapwood (S) and burning

Acetylated scots pine with heartwood (H) and pith.

Figure 4. Examples of acetylated Scots pine.

Figure 5. Slices of acetylated scots pine showing heartwood, sapwood, checking and "burning spots".
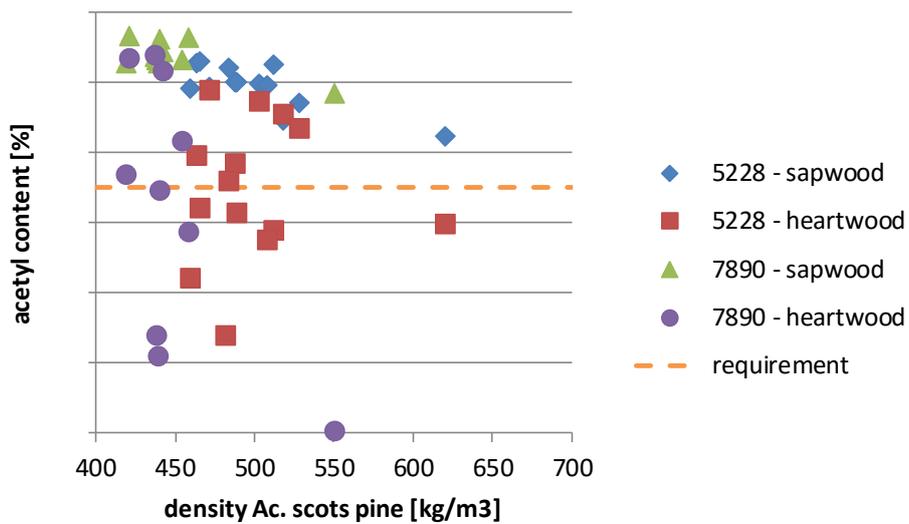
Figure 6. Relation between acetyl content and density for acetylated Scots pine sap- and heartwood for batch (7890) and batch (5228).

Batches 8495 and 8974

The results of batch 8495 were used to further develop a prediction model previously used to predict the result from the acetylation. The original model was based on measurement of different density values, and the developed model takes into account other characteristics of the wood material, based on X-ray measurements of the log, prior to cutting. This developed model is herein denoted "X-ray model". The calibration of the X-ray model included batch 8495 and, following this, the material acetylated in batch 8974 was used to verify the updated model. Batch 8974 consisted of 140 boards which were acetylated in a process originally developed for 32 mm thick Radiata pine boards.

When cutting the samples of batch 8974, after acetylation, it turned out that most samples (70%) were **not** well acetylated. Figure 7 gives an overview of the boards that were acetylated well (OK) and **not** well acetylated (NOT OK) in relation to the developed X-ray model. A comparison of the prediction model with the findings is also shown in Table 2. Only 25% of the specimens were predicted well. The results raise severe doubts on the correctness of the X-ray prediction model.

Table 2. Results of tests in relation to prediction model (based on 40 samples).

	Test results	
Model	**OK**	**NOT OK**
OK	12,5%	70,0%
NOT OK	5,0%	12,5%
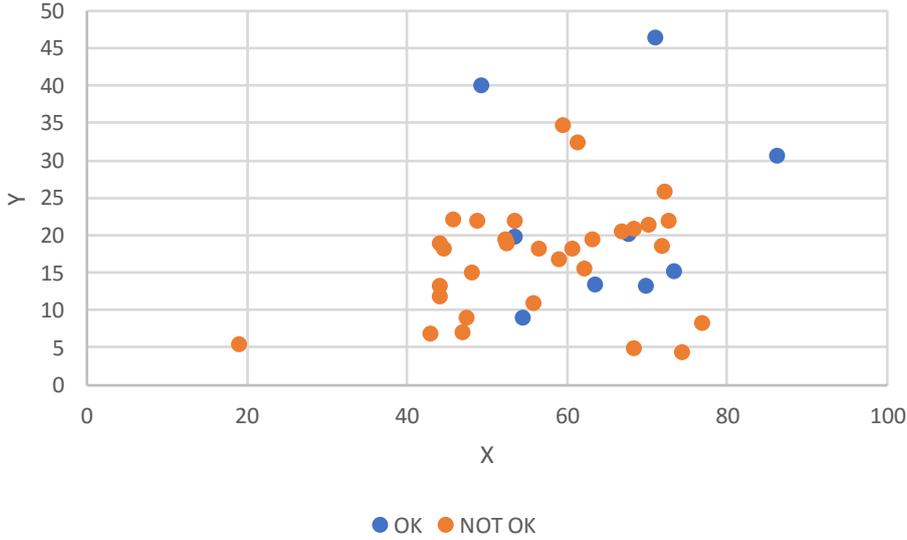
Figure 7. Results of acetylation of batch 8974 of samples acetylated OK and NOT OK in relation to X and Y (X-ray model).

2.3.2   Birch

The achieved acetyl contents comply to a minimum requirement (see Figure 8). Also the residual acetic acid content is low. Biggest issues are instead the internal checks and collapse. It should be noted that a pre-drying process and an acetylation process for radiata pine was used. If birch is of interest to investigate further, adjusted drying and acetylation processes are strongly advised.
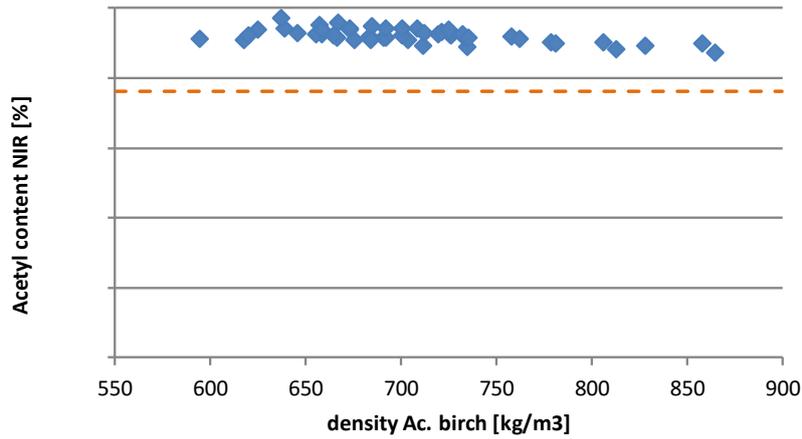
Figure 8. Relation between density of acetylated birch and acetyl content.

Figure 9. Acetylated birch (38xRW)

2.3.3 Birch veneer

The 3 mm rotary cut birch veneers from Koskisen were acetylated to sufficient high degrees, and also low residual acetic acid contents. A visual impression of the veneers is shown below. Some veneers have large checks. However, depending on the intended further use of the veneers, this might be of importance or not as regards structural behaviour. In terms of handling of the veneers, the severe cracking can be a problem, though. The outer surface of the multi-stacked veneers is discoloured due

to the acetylation process. This is mostly only limited to the top veneer sheet; the others are free from dark discolorations.

Figure 10. Acetylated birch veneers.

2.4 Conclusions

2.4.1 Scots pine

Previous research has showed consistent acetylation of sapwood. The acetylation of scots pine heartwood, however, is much more variable and insufficient consistent to make a guaranteed acetylated product. The same conclusion is drawn for acetylation of "young scots pine heartwood" (in the test circa 40% of the samples contained too low acetyl contents).

Within the project it has been tried to develop a model to predict success of acetylation based on X-ray measurements prior to cutting the log. The verification test of the model, however showed that a large percentage (70%) of the X-ray sorted scots pine heartwood, that was predicted to acetylate well, was **not acetylated well**. The results raise highly doubt on the correctness of the prediction model.

2.4.2 Birch

Sufficient high acetyl values and low residual acetic acid contents were found for acetylation of birch of 38 mm. Biggest issues are the internal checks and collapse. It should be noted that a pre-drying process and acetylation process for radiata pine was used. If birch is of interest to investigate further, adjusted drying and acetylation processes are strongly advised.

2.4.3 Birch veneer

The 3 mm rotary cut birch veneers from Koskisen was acetylated to sufficient high degrees, and low residual acetic acid contents. Several sheets had checking The acetylation process discolouration is mostly limited to the top veneer.

2.5 Recommendations

In respect to suitability for the acetylation process, only scots pine sapwood, and birch veneer seem interesting to explore further. Acetylation of solid dimensions of birch (38 mm thickness) will take much effort to improve drying and acetylation processes to minimise checking and collapse. Acetylation of scots pine heartwood seems to result in highly variable results and is not recommended to study further.

2.6 References

Alexander, J, Hague, J R B, Roberts, M, Imamura, Y and Bongers, F. (2014). The resistance of Accoya® and Tricoya® to attack by wood-destroying fungi and termites. *Proceedings IRG Annual Meeting*, IRG/WP 14-40658.

Beckers, E P J, Bongers, H P M, Van der Zee, M E and Sander, C (2003). Acetyl content determination using different analytical techniques. *European Conference on Wood Modification 2003*.

Beckers, E P J, Militz, H and Stevens, M (1994). Resistance of acetylated wood to basidiomycetes, soft rot and blue stain. *Proceedings IRG Annual Meeting*, IRG/WP 94-40021.

Bongers, F, Kutnik, M, Paulmier, I, Alexander, J. and Miltiz, H. (2015). Termite and insect resistance of acetylated wood. *Proceedings IRG Annual Meeting*, IRG/WP 15-40703.

Bongers, F., Roberts, M., Stebbins, H. and R. Rowell (2009). Introduction of Accoya® wood on the market – technical aspects. In: *Proceedings of the Forth European Conference on Wood Modification*. Stockholm, Sweden, pp. 301-310.

Goldstein, I S, Jeroski, E B, Lund, A E, Nielson, J F and Weaver, J W (1961). Acetylation of wood in lumber thickness. *Journal of Wood Chemistry and Technology* **11**(8): 363-370.

Larsson-Brelid, P and Westin, M (2010). Biological degradation of acetylated wood after 18 years in ground contact and 10 years in marine water. *Proceedings IRG Annual Meeting*, IRG/WP 10-40522.

Rowell, R M and Dickerson, J P (2014). Acetylation of wood. In: *Deterioration and protection of sustainable biomaterials*. Schultz, T P, Goodell, B, Nicholas, D D (eds.). ACS Symposium series 1158. American Chemical Society. ISBN 978-0-8412-3004-0.

Schwanninger, M, Stefke, B and Hinterstoisser, B (2011). Qualitative assessment of acetylated wood with infrared spectroscopic methods. *Journal of Near Infrared Spectroscopy* **19**(5), pp. 349–357.

Suttie, E D, Hill, C A S, Jones, D and Orsler, R J (1999). Chemically modified solid wood. I. Resistance to fungal attack. *Material und Organismen*, **32**: 159-182.

Takahashi, M, Imamura, Y and Tanahashi, M (1989). Effect of acetylation on decay resistance of wood against brown rot, white rot and soft rot fungi. *Proceedings IRG Annual Meeting*, IRG/WP 89-3540.

3 Durability of acetylated wood

Chemical modification of wood material through acetylation is well known to give the wood material an increased dimensional stability and improved rot resistance. To confirm this for the acetylated wood material used in this project durability testing was conducted both in laboratory scale and as a field test according to EN 252.

3.1 Laboratory tests

The laboratory tests have been performed on both young pine wood samples and birch wood. The acetylated samples have been taken from the material acetylated at Accoya in Arnhem. The durability test was conducted according to ENV 807, Figure 11, a test method where the test samples are exposed in unsterile soil and the fungal degradation is compared with unmodified control samples and preservative treated reference samples. The reference preservative is CCA (copper, chromium, arsenic at uptake levels of 3.78 and 9.21 kg/m^3. The levels correspond to NWPC Class AB and Class A (Use class 3 and 4).
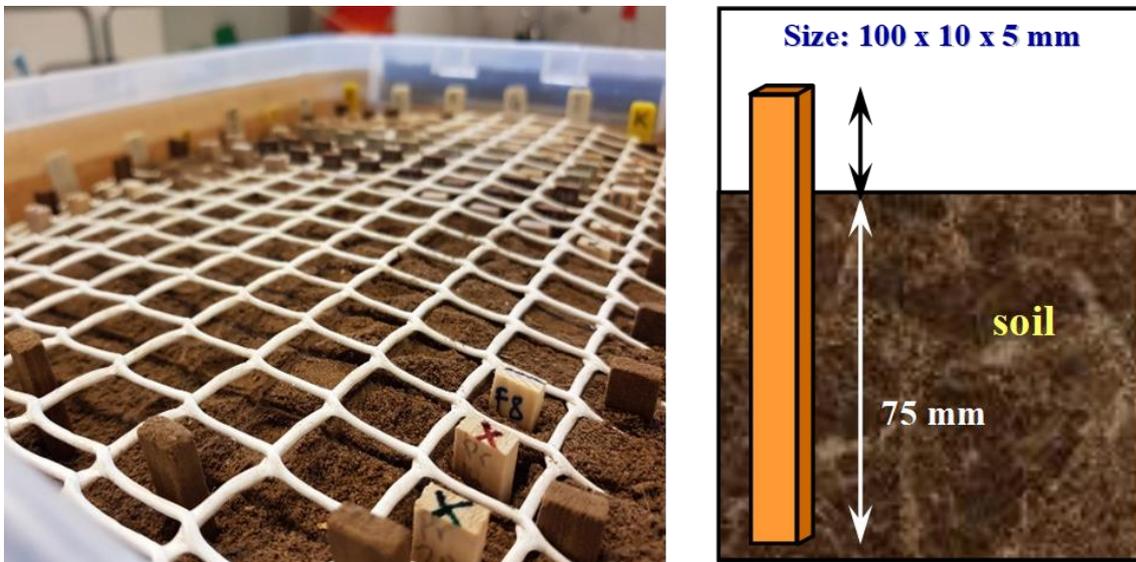
Figure 11. Test set -up for ENV 807.

Three test soils where prepared; one garden compost soil with neutral pH and soft rot is the prevailing degrading fungi, one brown rot soil with the dominating wood decaying organism being the brown rotting fungus, *Leucogyrophana pinastri*. The soil is meagre and sandy and has a very low WHC around 20-40%, and pH=5.2. The white rot soil is an acidic forest soil. All soils were sieved with a sieve with 4mm mesh and were moistened to approximately 95% of the water holding capacity (WHC) for both compost and brown rot soil. The soils were filled in 30-litre plastic boxes. All boxes had ventilated lids and were filled to a soil bed depth of approx. 15 cm.

Prior to the soil box test all samples were pre-aged according to EN 84 where the samples are water leeched in de-ionised water over a 14-days period. The water was exchanged 10 times during the incubation and the water volume in relation to specimen volume was 5:1. Dry-weights before and after leaching was recorded after oven-drying for 18h at 103ºC, after which the mass loss due to leaching was calculated. For all materials, a minimum of 8 specimens was used. The specimens were placed in the soil at a depth of approximately 7.5-8 cm with the identification mark above the soil. The decay

test was terminated after 16 weeks of incubation. Before harvest, control samples were checked for mass loss to insure sufficient decay. At the termination of the test, the specimens were carefully pulled out of the soil and cleaned before the wet-weights were recorded for calculation of moisture content. Dry-weights of all specimens after decay were recorded after oven-drying for 18h at 103°C, after which the mass loss was calculated.

Four specimens each of the test, control and reference specimens were used to determine the correction value (CV) for each soil, i.e. the amount of mass loss that is due to leaching of the samples to the soil. These samples were gamma sterilized with an absorbed dose of 34kGy to 34,3kGy. Also, the CV specimens were exposed for 16 weeks, cleaned and weighed according to the procedure described above. The mass loss values of the CV samples were then subtracted from the mass loss of the specimens in the decay test to obtain the corrected mass loss values, i.e. mass loss due to microbiological degradation.

3.2   Results

All acetylated test specimens lost similar amounts of mass during the leaching procedure as the reference samples. Standard deviation was below 10%. No sign of growth of inhibiting microorganisms, such as mould.

Table 3. Mass loss due to water leaching according to EN 84.

Material	Average mass loss (%)	Standard deviation (%)
Juvenile pine	1,0	0.2
Acetylated juvenile pine, low level	0.6	0.2
Acetylated juvenile pine, medium level	0.4	0.1
Acetylated juvenile pine, high level	0.3	0.0
CCA 9 kg	-0.4	0.0

Material	Average mass loss (%)	Standard deviation (%)
Acetylated birch	0.59	0.08
Acetylated birch plywood	1.35	0.13
Untreated birch plywood	3.49	0.14
CC 10 kg/m^3 reference	1.5	3.1
CCA 9 kg/m^3 reference	0.3	0.2
CCA 2 kg/m^3 reference	1.8	2.1

Results of the mass loss in the soil block test are presented in Figure 12 and Figure 13. As expected, low mass losses were obtained both in the trial with young pine and in the trial with birch. Mass losses for the samples exposed in the Forest soil (white rot soil) will not be presented due to low activity in that soil revealed by much to low mass loss of the controls. Figure 12 shows that for acetylated young pine the mass losses were very low, even for a low acetylation treatment level (acetyl content: 14-15%) the mass loss did not exceed 2,5% and for the high level (approved Accoya standard) the mass loss was less than 0,5%. The same low mass losses were obtained for the birch wood and for birch plywood no mass loss due to fungal attack occurred. The mass loss of the unmodified test samples shows good fungal activity in the soils with figures exceeding 35%. For the acetylated test samples the mass loss was in the same range of less, when compared with the preservative treated wood. In conclusion, the tests confirm a major increase in fungal resistance gained by acetylation.
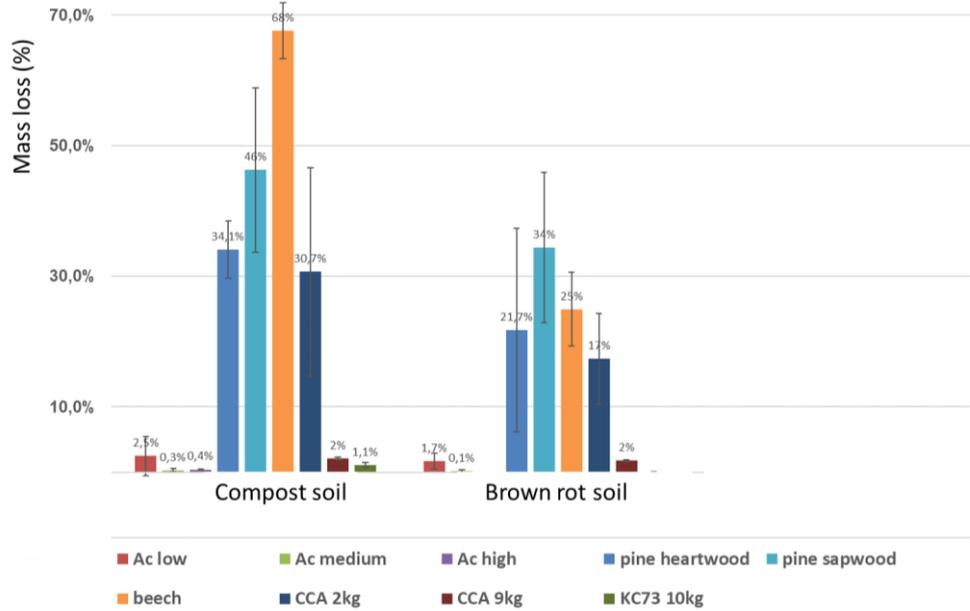
Figure 12. Mass loss (%) for acetylated young pine compared with preservative treated references and unmodified control samples
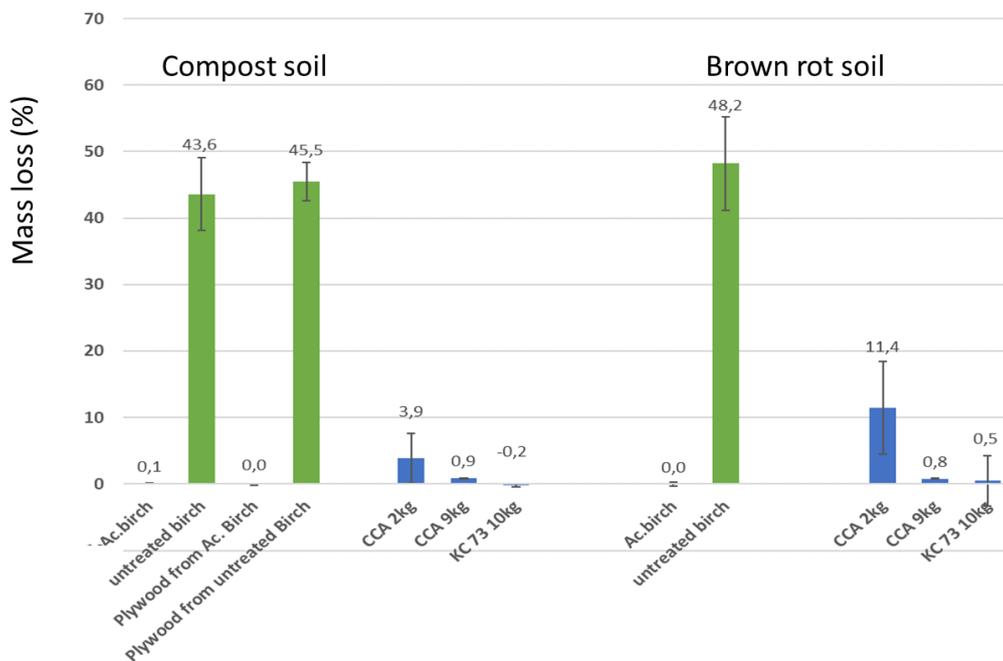

Figure 13. Mass loss (%) for acetylated birch wood and birch plywood compared with preservative treated references and unmodified control samples.

3.3 Field tests

Acetylated young pine and birch wood has also been exposed in ground contact in a field test. The test has been conducted according to EN 252 (Figure 14).

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Tillstånd	Bedömning	Rötindex
Friskt	0	0
Svaga angrepp	1	25
Måttliga angrepp	2	50
Svåra angrepp	3	75
Utdömt	4	100

Figure 14. Field test according to EN 252.

As in the laboratory test, the acetylated wood was evaluated together with preservative treated reference test stakes and unmodified control sakes. The size the test stakes are 25 x 50 x 500 mm and 10 stakes for each treatment were inserted in the field to 2/3 of its length. The test stakes are assessed once a year, using the grading from 0 to 4, where 0 is sound- no decay, and 4 is very severe decayed (stake rejected). By adding the grading of decay for the stakes of each group and dividing the sum by the number of stakes, the index of decay is calculated by multiplying by 25. The test was started in October 2018 and as expected, no sign of decay was detected on any of the acetylated test stakes. For unmodified young pine and birch wood a slight fungal attack could be observed after 2 years exposure.

To put the field test in perspective, results from an earlier field test conducted in the test field in Simlångsdalen, Sweden. The test was set out in 2003 and the size of this samples were 30 x 30 x 500 mm. The index of decay is presented in figure 4. After 17 years the index of decay is still 50%, which means that on an average only half of the test stakes have failed. In general, birch is considered a very easily degradable wood specie and that is also shown in the figure where all test stakes of the unmodified birch wood have failed after 4 years exposure.
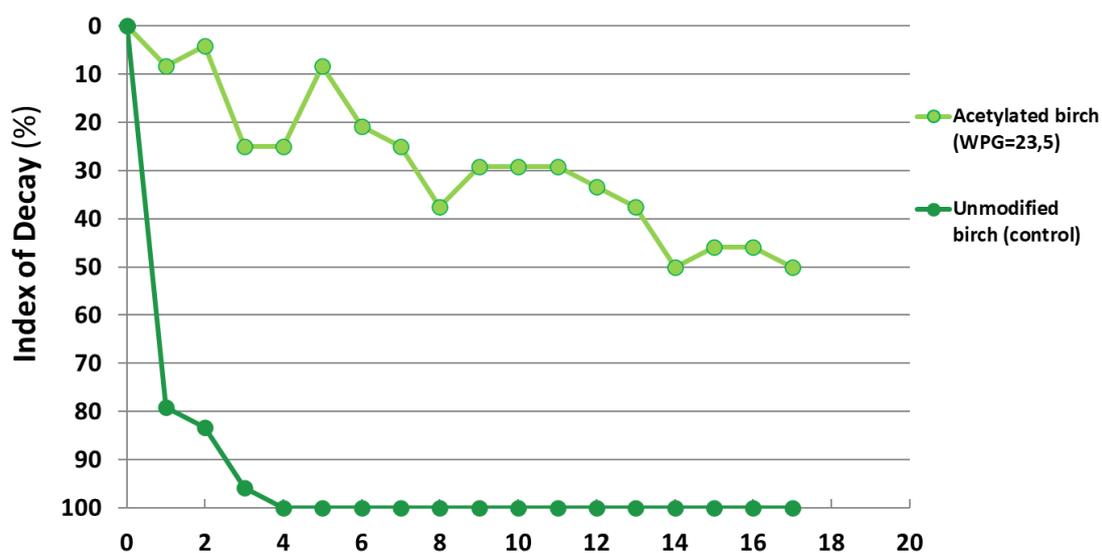
Figure 4. Index of Decay for acetylated birch after 17 years exposure in Simlångsdalen, Sweden.

4 Gluing of acetylated wood

4.1 Overview

This study was performed by partner AkzoNobel and focused on acetylated birch and acetylated juvenile pine with the specific aim to investigate the possibility to glue acetylated wood. The study included contact angle measurements with water and investigations of the gluing performance of commercially available AkzoNobel Adhesive systems MUF, PUR, EPI and PRF on acetylated birch and acetylated juvenile pine. The study was divided into two parts: 1. *Screening Adhesive Systems* and 2. *Parameter Optimization*. In the first part of the study, commonly used gluing parameters were investigated for each respective adhesive system and application. In the next part of the study the gluing parameters were optimized for acetylated wood. Glued beams were evaluated with a chisel test (% fibre tear directly after pressing) and delamination according to EN14080, Annex C, Method B.

4.2 Background

4.2.1 Adhesives

Formaldehyde- and Isocyanate-based adhesive systems are two groups of thermosets commonly used for gluing load-bearing timber constructions.

MUF, Melamine Urea Formaldehyde, adhesives are formed through a condensation reaction where melamine and urea react with formaldehyde respectively. The resulting units reacts with each other in a polycondensation, creating a cross-linked network. It is a 2-component system that cures with an acidic hardener, Figure 15. [1], [5].

PRF, Phenol Resorcinol Formaldehyde, adhesive has a similar co-condensation reaction to that of MUF. Phenol and resorcinol react with formaldehyde respectively into a resin. The resin cures as a 2-component system together with formaldehyde as hardener, Figure 16. [2].

PUR, one component polyurethane, consists of cross-linked reactive NCO-groups on a pre-polymer back-bone. The curing is initiated by NCO-reaction with ambient moisture. Figure 17. [3].

EPI, Emulsion Polymer Isocyanate, is a water-based 2C mixture of emulsion polymer/dispersion and an isocyanate hardener. Prior to gluing, the two components are mixed, and the dispersion is cross-linked via isocyanates. [4]
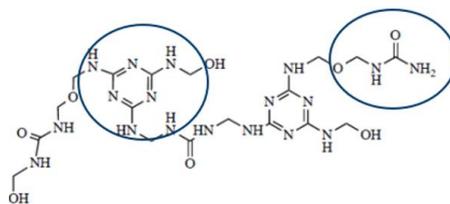

*Figure 15. MUF, blue rings mark initial reactants melamine & urea.*
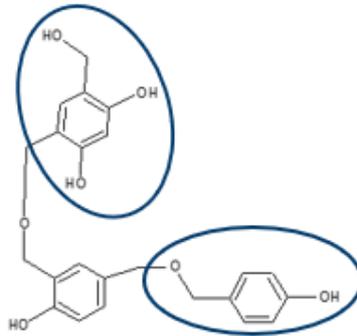
Figure 16. PRF, blue rings mark initial reactants phenol and resorcinol
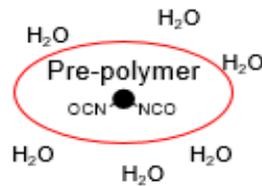
Figure 17. PUR, red ring marks the pre-polymer which cures in contact with moisture
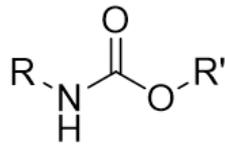
Figure 18. EPI/Urethane (Isocyanate (NCO) reacts with primary alcohols (mainly in PVA) to form urethane linkages).

4.2.2 Gluing

Adhesion is a process of two substrates bonding together through their surfaces by intermolecular forces. There are several theories of the course of adhesion.

The *Adsorption theory* is most frequently used for wood adhesives. It states that the adhesion between an adhesive and a wood substrate is caused by secondary forces between the molecules present in the two materials. van der Waals forces, electrostatic interactions and hydrogen bonds all contribute to the adhesion.

The *Mechanical Interlocking theory* is a mechanism believed to be applicable in most cases, even though its contribution to adhesion is thought to be limited. The adhesion occurs through penetration of the adhesive into pores and cavities of the wood surface. The degree of interlocking is significantly dependent on the micro- and macroscopic surface roughness of the wood. Regarding thermosetting wood adhesives, it is desirable to achieve a certain degree of penetration into the substrate since adhesive curing in the cavities is believed to enhance the strength of the glue joint.

The *Covalent Chemical Bonding theory* states that a reaction between the adhesive and wood substrate results in formation of chemical bonds between the two. This process is believed to be relevant for melamine formaldehyde (MF) and urea formaldehyde (UF) resins. [5,6] In order to enable

a sufficient adhesion, the adhesive needs to fully wet the surface of the wood, fill all cavities and thus displaces potential air pockets. The wetting enables intermolecular interactions to occur between molecules of the wood and the adhesive. The wettability is influenced by adhesive properties such as viscosity, molecular weight and molecular weight distribution. Additional important parameters are the porosity and hardness of the wood substrate. The wetting and adhesion will in turn influence the final mechanical properties of the cured glue joint. [5,7,8]

4.2.3   Acetylated Wood

Acetylation of wood is performed by impregnation of acetic anhydride which reacts with wood polymers in an esterification reaction of the hydroxyl group resulting in acetic acid as a by-product.

$$\text{WOOD–OH} + \text{CH}_3\text{–C(=O)–O–C(C=O)–CH}_3 \rightarrow \text{WOOD–O–C(=O)–CH}_3 + \text{CH}_3\text{–C(=O)-OH}$$
<div align="center">Acetic anhydride   Acetylated wood   Acetic acid</div>
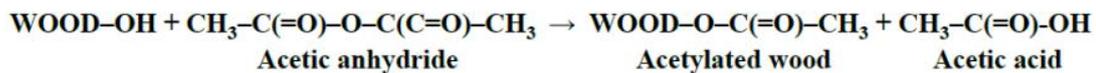
Figure 19. Acetylation of wood

The acetylated wood is more hydrophobic resulting in improved biological resistance, dimensional stability and hardness. The surface properties of the wood are important for the gluing performance and will affect wetting, adhesion/penetration, chemical interactions and bonding. [9]

4.3   Experimental

The gluing performance of commercially available AkzoNobel Adhesive systems MUF, PUR, EPI and PRF on acetylated birch and acetylated juvenile pine was investigated. Additionally, the surface properties of the acetylated wood were studied further by means of contact angle measurements with the aim to increase understanding of the gluing performance on acetylated wood. The study was divided into two parts: 1. Screening Adhesive Systems and 2. Parameter Optimization

4.3.1   Methods

Contact angle of water on wood
Contact angle measurements were performed on wood samples of acetylated juvenile pine, Juvenile pine and standard pine. The wood pieces were treated by an abrasive belt grinder roughly 30 min prior to contact angle analysis. 1 drop of water was applied to the substrate pieces at 5 different spots by letting a drop fall down roughly 1 cm. The wetting of the substrate was recorded for 51 seconds using a video camera. The initial contact angle (after about 100 ms) and the contact angle 51 seconds after "touch down" were evaluated. The initial contact angle is related to surface properties of the wood before wetting, while that after 51 seconds is also affected by penetration.

Gluing and physical testing
The gluing performance of commercially available AkzoNobel Adhesive systems MUF, PUR, EPI and PRF on acetylated birch and acetylated juvenile pine was investigated. Non-acetylated birch and non-acetylated juvenile pine was used as references. Prior to gluing, the wood having the dimensions 285×100×25 mm³, was conditioned for five days at reference conditions 20°C/65% RH. The adhesive and hardener were mixed together prior to gluing and the adhesive system was applied manually with a spatula onto one side of each wood lamella. The gluing parameters used are in accordance with the European standard EN14080, Annex C, Method B. In the first part of the study (*1. Screening Adhesive Systems*) gluing parameters (adhesive: hardener mixing ratio, assembly time (TA) and glue spread (GS)) commonly used for the specific adhesive system and application was tested. In the next part of the study (*2. Parameter Optimization*) the gluing parameters were further investigated. 5 lamellas were

assembled into a beam and pressed with 8 MPa at 20°C overnight. After pressing, the fibre tear of each glue joint was investigated by chisel testing. Approved results are obtained when >75% of the surface is covered with fibre. The beam was also subjected to a delamination test including water soaking followed by drying according to EN14080 Annex C, Method B. The delamination of each glue joint was then calculated. Approved results are obtained when the delamination for each glue joint and the average delamination of the beam is <4%.

4.3.2   Samples and Parameters

In Table 4 all samples used in the study are presented, and in Table 5 the different gluing parameters used can be found.

Table 4. adhesives and hardeners used in this study.

Adhesive Type	Hardener Type
MUF	Acidic
EPI1	Isocyanate
EPI2	Isocyanate
PRF	Formaldehyde
PUR1*	-
PUR2*	-

One component PUR.

Table 5. gluing parameters used in this study.

Adhesive	Mixing Ratio (A:H)		Assembly Time (TA) [min]			Glue Spread (GS) [g/m2]	
MUF	Normal		Normal			Normal	
EPI1	Normal		Short		Normal	Low	High
EPI2	Normal		Long			High	
PRF	High	Low	Short	Normal	Long	Normal	
PUR1	Normal		Long		Short	Normal	
PUR2	Normal		Long			Normal	

4.4   Results and Discussion

4.4.1   Contact angle of water on wood

The initial water contact angle was clearly higher on the acetylated juvenile pine compared to the reference pine, Table 6. The contact angle decreased gradually to on average 61° after 20 seconds and to 57° after 51 seconds for the acetylated wood. For the reference wood the contact angle decreased dramatically with time and quickly penetrated the wood. The course of events was perhaps slightly slower for the juvenile pine compared to the standard pine. Thus, the acetylation has a very large effect on the wetting properties of this type of wood.

Table 6. Water contact angles on acetylated juvenile pine, juvenile pine and standard pine.

	Water contact angle (°)		
Sample	**Initially**	**After 20 s**	**After 51 s**
Acetylated juvenile pine	76±3	61±2	57±1
Juvenile pine ref	52±1	0	0
Standard pine	56±7	0	0

4.4.2   Screening Adhesives Systems

In the first part of the study, the gluing performance was investigated with commonly used gluing parameters for each specific adhesive system. The results of EN14080, fibre tear and delamination on acetylated juvenile pine, can be found in Figure 20 and Figure 21. As can be seen, the fibre tear is approved for all systems except for MUF which are just below the limit. Indications of reduced performance of MUF on acetylated wood is further verified by the results from the delamination test. MUF has significantly higher delamination compared to the other adhesive systems. MUF and EPI fails the test, while PRF and PUR pass the requirements. Correspondingly results on acetylated birch can be seen in Figure 22 and Figure 23. The results are in generally lower for all adhesives systems when gluing on acetylated birch compared to acetylated juvenile pine. Only PUR is just below the limit to pass the fibre tear requirement and none of the adhesive systems fulfils the delamination requirement. The difference in gluing performance on juvenile pine and birch could be assigned to the higher density of birch, affecting the penetration and glueability. The initial test results indicate that managing a way of gluing acetylated wood with MUF probably require major modification of the adhesive. Hence, MUF will be excluded from further study. However, it is possible that satisfying results can be obtained with PRF, EPI and PUR if gluing parameters are optimized. See summary of concluding results in Table 7.
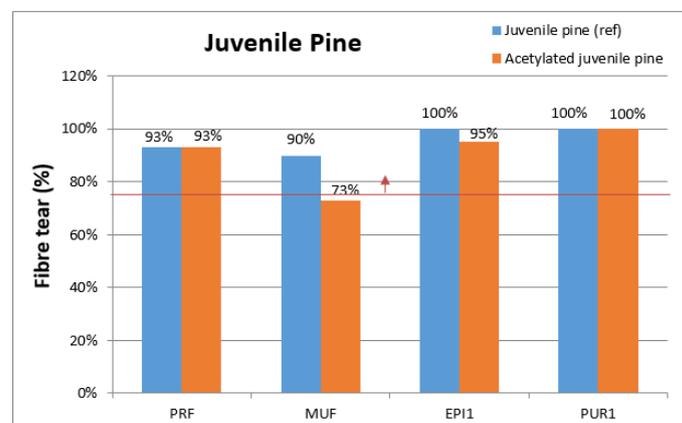
Figure 20. Fibre tear (%) for each investigated adhesive system glued on acetylated juvenile pine (orange) and reference juvenile pine (blue). Internal requirement >75%.
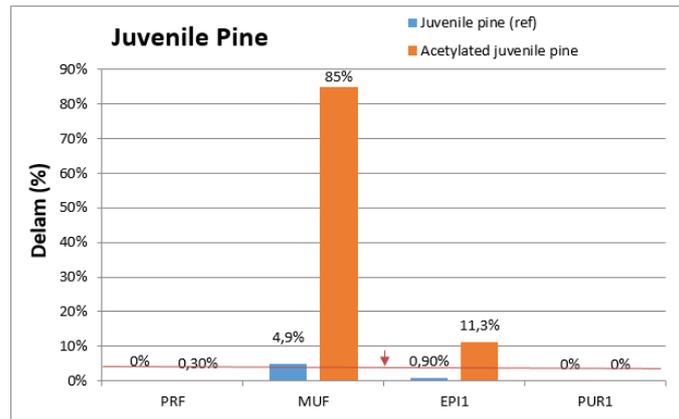
Figure 21. Delamination (%) for each investigated adhesive system glued on acetylated juvenile pine (orange) and reference juvenile pine (blue). Requirement acc. to EN14080 <4%.
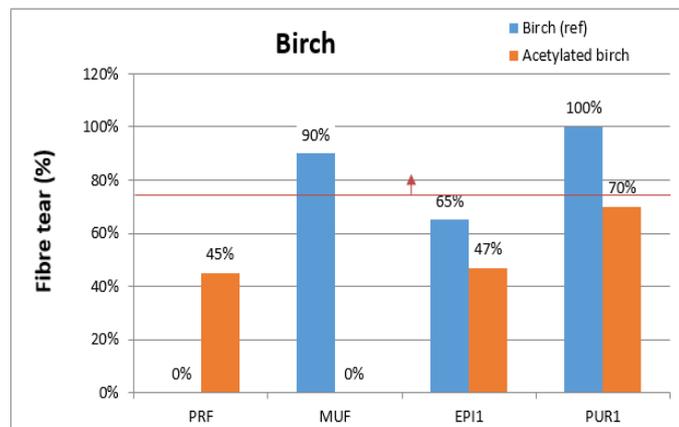

Figure 22. Fibre tear (%) for each investigated adhesive system glued on acetylated birch (orange) and reference birch (blue). Internal requirement >75%.
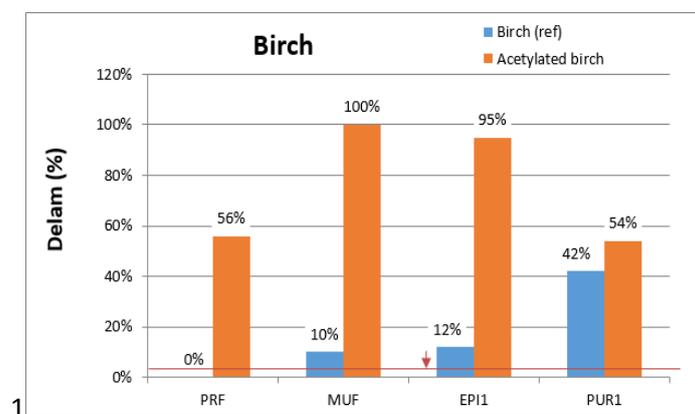
*Figure 23. Delamination (%) for each investigated adhesive system glued on acetylated birch (orange) and reference birch (blue). Requirement acc. to EN14080 <4%.*

Table 7. Summarizing results of Fibre tear and delamination according to EN14080.

Adhesive system	Pine		Birch	
	Ref	Acetylated	Ref	Acetylated
MUF	Approved	Not approved	Not approved	Not approved
EPI	Approved	Not approved	Not approved	Not approved
PRF	Approved	Approved	Not approved	Not approved
PUR	Approved	Approved	Not approved	Not approved

4.4.3   Parameter Optimization

From the first part of the study it was concluded that it is too farfetched to obtain satisfying gluing's with the investigated MUF. It is also believed to be a greater challenge to glue on acetylated birch compared to acetylated juvenile pine. Therefore, MUF and acetylated juvenile pine was excluded from further study and is therefore not included in part 2 *parameter optimization*. In this second part of the study, the gluing parameters (mixing ratio, assembly time and glue spread) were varied in order to find suitable gluing conditions on acetylated birch for each adhesive system. The results can be seen in Figure 24 and Figure 25. For PUR1, a long and short assembly time was tested. Additionally, two different PUR, PUR1 (faster curing) and PUR2 (slower curing), were investigated in order to find trends. Both fibre tear and delamination seem to be somewhat better with short assembly time for PUR1 compared to PUR2. Overall, the PUR adhesives does not fulfil the requirements. However, there are indications that the gluing results with PUR could be improved. EPI was further studied both by changing the glue spread and the assembly time. Additionally, two different EPI, EPI1 (faster curing) and EPI2 (slower curing), was tested together with long and short assembly times to find trends. Double-sided application was made to optimize adhesion. None of the glue-ups passed the test and no clear trend can be seen. PRF was initially glued with low mixing ratio and normal assembly time and showed promising trends. However, due to indications of pre-curing, a long and short assembly time was evaluated together with increased mixing ratio in order to find an optimum. PRF indicates to be the most robust adhesive system and shows best gluing performance on acetylated birch. The changes made in assembly times does not have a significant impact on the gluing results. However, long assembly time seem promising.

To summarize:

- PUR and EPI had low fibre tear and high delamination compared to PRF.
- PRF is the only system that passed the delamination test and has promising fibre tear.
- High fibre tear was obtained for all references compared to for acetylated birch.
- Least delamination was obtained from glue-ups with PRF both on acetylated birch and the reference birch.

See summary of concluding results in Table 8.
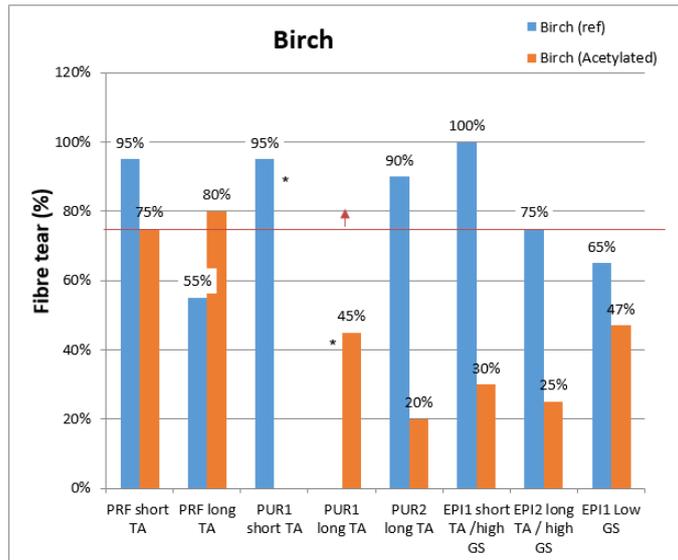
*Figure 24. Fibre tear (%) for each investigated adhesive system and parameter, glued on acetylated birch (orange) and reference birch (blue). Internal requirement >75%. * No corresponding gluing on reference birch or acetylated birch.*
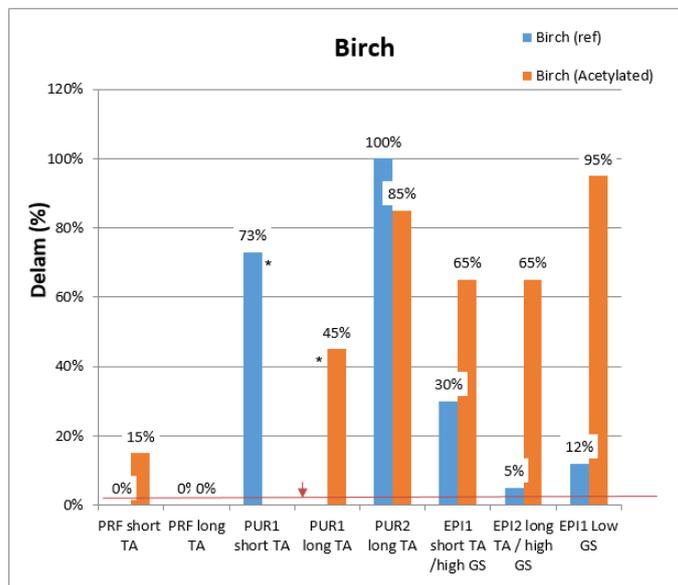
*Figure 25. Delamination (%) for each investigated adhesive system and parameter, glued on acetylated birch (orange) and reference birch (blue). Requirement acc. to EN14080 <4%. * No corresponding gluing on reference birch or acetylated birch.*

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Table 8. Summarizing results of fibre tear and delamination according to EN14080.

System & parameter	PRF Short TA	PRF Long TA	PUR1 Short TA	PUR1 Long TA	PUR2 Long TA	EPI 1 Short TA/high GS	EPI 1 Low GS	EPI 2 long TA
Ref Birch	Not approved	Approved	Not approved	Not approved	Not approved	Not approved	Not approved	Not approved
Acetylated birch	Not approved	Approved	Not approved	Not approved	Not approved	Not approved	Not approved	Not approved

4.5   Conclusions

The following main conclusions are drawn from this study

- Acetylated wood is generally difficult to glue.
  - Acetylated birch (hardwood) more difficult than acetylated pine (softwood).
  - Acetylated is more hydrophobic than ref wood.
- **PRF** indicates to be the most robust adhesive and has the best gluing performance on acetylated birch.
  - The tested gluing parameters does not have a significant impact on the results. However, it seems that some improvements could be obtained by optimization.
  - **Long assembly time** for PRF seem promising.

4.6   Further work

The following methods are recommended to further investigate:

- Block shear test
- Beam dimension stability and weight
- Contact angle of water on adhesive applied on wood
- Penetration of different adhesive systems (SEM/IR)

According to the study performed at AkzoNobel Adhesives AB the glueability on acetylated wood is challenging. The PRF adhesive seem to be the only robust alternative. At the time of performing this study, it will be a great challenge for acetylated wood to obtain an approval for load-bearing constructions. The available test standards aim to expose the glue joint to forces caused by swelling and shrinkage of the wood material. Hence, dimension stable acetylated wood results in a deceptive evaluation of strength of the wood adhesive. It is therefore believed that an important step in the process of implementing acetylated (birch) wood on the market is to develop a test method for hardwood, e.g. a block shear test with a climate cycling and/or test pieces for EN302-1, gap joint tests. Furthermore, the brittleness of acetylated wood will be an issue for the load-bearing application since the wood should not be weaker than its application. Hence, from a test standard and application point of view the production of non-loadbearing particle boards from acetylated wood is less of a challenge.

4.7   References

[1]   A Nuryaw, A. P. Singh och B.-D. Park, "*Swelling Behavior of Cured Urea-Formaldehyde Resin Adhesives with Different Formaldehyde/Urea Mole Ratios*," The Journal of Adhesion, 2014, pp. 2-3.

[2]   A.H. Conner, M.S.H. Bhuiyan, "*Module in Materials Science and Materials Engineering*", 2017.

[3]   "*Encyclopedia of Polymer Science and Technology*", John Wiley & Sons Inc, 2011, pp. 2.

[4]   K. Grøstad and A. Pedersen, "*Emulsion Polymer Isocyanates as Wood Adhesive: A Review*", Journal of Adhesion Science and Technology 24 (2010), pp. 1357–1381.

[5]   A. Pizzi, "*Advanced Wood Adhesives Technology*", New York: Marcel Dekker, Inc, 1994, pp. 1-14,19-22, 41-43, 68-71, 77-78.

[6]   S. Ebnesajjad, "*Adhesives Technology Handbook*", 2nd Edition, New York: William Andrew Inc, 2008, pp. 3-11, 24-25, 47-55,73-74, 184, 192-194.

[7]   Y. Li och S. Ren, "*Building decorative materials*", Cambridge: Woodhead Publishing, 2011, pp. 326-327, 330-331.

[8]   J. Bently och G. Turner, *"Introduction to Paint Chemistry and Principles of Paint Technology"*, 4th ed, London: Chapman & Hall, 1997, p. 95.

[9]   G. Mantanis, "*Chemical Modification of Wood by Acetylation or Furfurylation: A Review of the Present Scaled-Up Technologies*", Greece: BioResources, 2017, pp.4479-4481

5 Mechanical characterisation

5.1 Background and aim

The aim of the material characterisation was to quantify the influence of the acetylation on mechanical properties relevant for structural applications. Although many previous studies have focused on mechanical properties of acetylated wood, little is known in terms of strength in tension perpendicular to grain and fracture behaviour in terms of specific fracture energy. The results from the research on characterisation of the mechanical properties of the material are described in more detail in [1] – [7].

5.2 Test methods and overview of tests performed

The tests performed in this project included standard measurements for the determination of wood density and moisture content (MC) by the oven-dry method. In addition, the tests aimed at determining:

- Tensile strength perpendicular to the grain (pine)
- Modulus of elasticity parallel to the grain (pine)
- Specific fracture energy in tension perpendicular to the grain (pine and birch)
- Sorption isotherms, i.e. determination of equilibrium moisture content (EMC) for various relative humidities (RH), (pine)

5.3 Fracture energy, tensile strength perpendicular to grain and MOE along grain

Untreated and acetylated Scots pine and birch was tested. The pine material originated from young logs, aged 30 – 40 years. The acetyl content of the modified specimens was approximately 20%. Clear wood specimens, consisting of either heartwood or sapwood, were extracted and conditioned to equilibrium at a relative humidity of 60% and a temperature of 20°C. The specific fracture energy was determined by tests using the Single Edge Notched Beam (SENB) specimen in three-point-bending, seeFigure 26.

Figure 26. Three-point SENB specimen used for determination of specific fracture energy.

The modulus of elasticity along the grain and the tensile strength perpendicular to the grain were also determined by the use of specimens loaded in compression and in direct tension, respectively, see Figure 27.

26
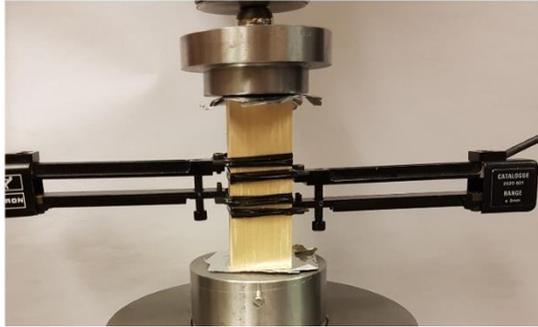
Figure 27. Left: Specimen mounted in testing machine for compression test to determine MOE along grain. Right: Direct tension test to determine tensile strength perpendicular to grain.

5.4   Results and conclusions

The main findings are presented in Table 9. A significant decrease of the fracture energy for acetylated specimens, in the order of 35–50%, compared to the unmodified specimens, was found for both pine and birch. No effect of the acetylation process on the MOE or the tensile strength was found (pine).

Table 9. Main findings from [1]and [6], results relate to wood conditioned at 20°C and 60% RH.

	Control			Acetylated		
	Fracture energy (J/m^2)	Tensile strength (MPa)	MOE (MPa)	Fracture energy (J/m^2)	Tensile strength (MPa)	MOE (MPa)
Pine sapwood, [1]	339	2.7	12 600	169	2.5	11 900
Pine heartwood, [1]	249	-	-	158	-	-
Birch, [6]	427	-	-	194	-	-

In [2] further tests with Scots pine and birch regarding the fracture energy are reported. The investigation aimed at clarifying the effect of moisture content on the specific fracture energy. The main findings are reported in Table 10, from which it can be concluded that the influence of acetylation on fracture energy is most severe at around 75% RH, where the reduction was around 50%. In addition, it can be concluded that the reduced fracture energy of acetylated wood at a given RH, only in part is due to the lower MC at that RH.

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Table 10. Main findings from [2], in terms of specific fracture energy (G_F) and moisture content (MC) for pine and birch conditioned at 20°C and various RH levels, including also oven dry (OD) and water saturated (WS) samples.

Species	Property	Control					Acetylated				
		OD	RH=20	RH=75	RH=97	WS	OD	RH=20	RH=75	RH=97	WS
Pine	G_F (J/m²)	220	242	387	348	283	165	175	197	256	299
	MC (%)	1.7	4.9	13.7	26.1	37.4	0.4	1.3	4.6	8.3	20.3
Birch	G_F (J/m²)	198	243	460	336	272	172	183	218	279	364
	MC (%)	1.2	4.1	13.6	29.8	30.9	0.3	1.5	5.6	10.2	17.3

The general conclusions drawn from the studies on the mechanical characteristics are that for acetylated wood:

- a significantly lower equilibrium moisture content was observed
- a significantly lower fracture energy at equal RH was observed (35% to 50% lower)
- no significant decrease in tensile strength perpendicular to the grain was observed
- no significant decrease in modulus of elasticity parallel to the grain was observed
- the influence of RH on fracture energy was most severe at around 75% RH
- the reduced fracture energy of the acetylated wood is mainly, but not solely, explained by the lower moisture content at equal RH

5.5   References

[1] Forsman, K., Serrano, E., Danielsson, H., Engqvist, J. Fracture characteristics of acetylated young Scots pine*. European Journal of Wood and Wood Products volume 78, pp. 693–703(2020)*

[2] Forsman, K., Fredriksson, M., Serrano, E., Danielsson, H. Moisture-dependency of the fracture energy of wood: A comparison of unmodified and acetylated Scots pine and birch. Accepted for publication in *Holzforschung*.

[3] Forsman, K., Danielsson, H. and Serrano, E. Fracture Characteristics of Acetylated Scots Pine - An experimental and numerical investigation. CompWood 2019, 17 – 19 June 2019, Växjö, Sweden.

[4] Forsman, K., Serrano, E. and Danielsson, H. Modified wood in outdoor load-bearing structures. Oral and poster presentation. Young researcher's program of the 2019 Marcus Wallenberg Prize events, 7-8 October, 2019.

[5] Forsman, K., Serrano, E. and Danielsson, H., Fracture characteristics of acetylated scots pine and birch, Oral presentation at 15th Annual Meeting of the Northern European Network for Wood Science and Engineering, Lund, 9-10 October 2019

[6] Lai, G. and Plönning, S. (2019) Fracture characteristics of acetylated birch - Experimental and numerical studies, Master's thesis, Report TVSM-5234, Division of Structural Mechanics, Lund University, Lund Sweden. http://lup.lub.lu.se/student-papers/record/8990739

[7] Forsman, K., Fredriksson, M., Serrano, E., Danielsson, H. Moisture-dependency of the brittleness of acetylated wood, Oral presentation at the 16th Annual Meeting of the Northern European Network for Wood Science and Engineering, Helsinki, 1-2 December 2020.

6 Corrosion – Notes on research needs

6.1 General

Thanks to the contemporary presence of representatives of different parts of the wood sector, an interdisciplinary exchange involving wood chemistry, biology, engineering, and production has been possible. This interdisciplinary approach finds its natural application in wood corrosion where wood structure, wood composition, interactions with the environment, and wood decay simultaneously act.

The project acted as a catalyst for a mixture of subjects which ended with a multidisciplinary approach to corrosion and as a basis for future development on wood corrosion studies.

Fasteners' corrosion is mostly affected by wood humidity as is the case of wood decay, and the correct approach is designing aiming for durability. Regarding wood decay, wood treatment aggressiveness, and the influence of design to control the EMC of wood, some literature can be found; a proof of the effectiveness of wood free volume reduction and maximum water uptake reduction to reduce/avoid corrosion is still missing.

6.2 On corrosion

Corrosion is a *spontaneous* and *natural* phenomenon that normally occurs in its simplest form every time that a metal, electrolyte, and oxygen are present simultaneously.

This is valid for all metals, including stainless steel or aluminium. The difference lies in the corrosion speed which for some metals is far lower than for others. For example: The speed of corrosion of stainless steel is lower than that of zinc.

Four mechanisms take place and must act simultaneously:

- Anodic reaction (where the metal corrodes)
- Cathodic reaction (where corrosion products accumulate)
- Electron transport in the electrolyte. The electrolyte in wood is the water.
- Electron transport in the metal

The choice of material (of the connector) is not the only factor influencing the screw durability towards corrosion. Also, the environmental conditions might affect the corrosion resistance of a connector. For example: Aluminium is proven to have very high durability in neutral environments but quite low in basic ones (pH>7). Something similar may happen inside the wood, where if some conditions are present simultaneously, it might become a quite aggressive environment.

6.2.1 Corrosion in wood

Since it is very difficult to simultaneously manage all the factors influencing corrosion (humidity, oxygen, metal surface, wood acidity, tannins, pollutants…) the method to avoid corrosion is to keep the moisture content under the threshold of minimum effect. This EMC threshold has been investigated by researchers and is the value (MC%) below which no corrosion can take place because of the absence of electron transport in the electrolyte (water).

This moisture content threshold is approximately 16%. This means that above this value a certain "amount" of corrosion may be present, below it cannot. Between 16% and 20% corrosion is quite low, above 20% corrosion starts to be significant (note that above 20% MC also rot decay can take place in the wood).
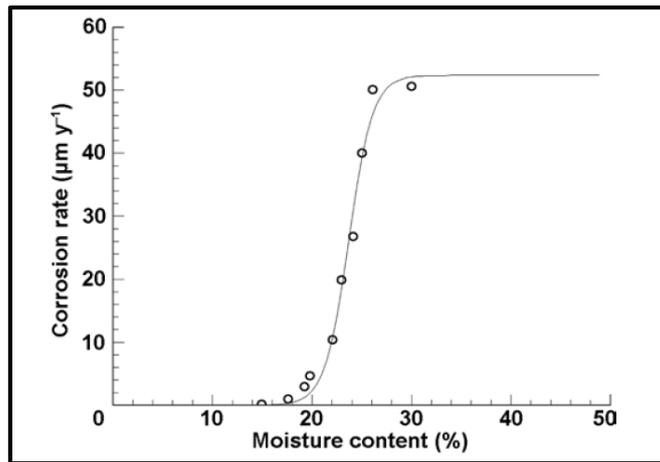
Figure 28 – Corrosion model in this study derived from Short and Dennis (1997) and Zelinka and others (2008)

From Figure 28 we understand the importance of keeping the wood dry. Not only for wood decay reasons but also to eliminate corrosion of metallic fasteners.

6.3   Building for durability:

Generally speaking, we can say that all design good practices which aim to enhance the durability of the wood are also ameliorative for fasteners' durability.

Fortunately, in service-classes 1 and 2 the MC is generally lower than 20% (according to EN 1995-1-1). In SC2 sometimes the MC may be higher than 16% and that's why a coating is required for the connectors.


☐ Service class 1
☐ Service class 2
☐ Service class 3
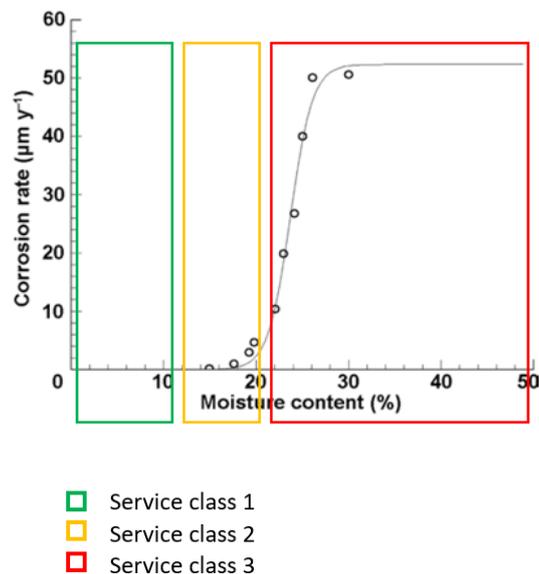
Figure 29 – Corrosion model in this study derived from Short and Dennis (1997) and Zelinka and Others (2008)

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6.4   Conclusions and need for further studies

Within the project, a corrosion approach to wood design was proposed, integrating EC5 prescriptions, Samuel Zelinka's studies, and design for durability prescription as the one indicated in DIN 68800-2.

- Tests on acetylated wood aiming to demonstrate if a correct design and the maximum EMC reduction below the 16% threshold are sufficient to avoid corrosion even if the treated wood can be even more aggressive than untreated wood are needed.
- Acetylated wood offers the possibility to investigate if water (more than 16% EMC) could really be considered as the necessary condition to have corrosion or other parameters as the acidity can be a sufficient condition for corrosion even in case of low moisture content.

7 Finger joints, hybrid components, plywood and surface-modified acetylated birch

7.1 Finger-jointing of acetylated Scots pine using a conventional MUF resin

The objective of this work, see [1] was to study finger-jointing of acetylated Scots pine (Pinus sylvestris L.) using a conventional melamine urea formaldehyde (MUF) adhesive. Two different types of acetylated pine specimens were investigated, acetylated pine sapwood (APS) and acetylated juvenile pine (AJP), the latter originating from young forest thinning trees (ca 20-30 years). The goal was to evaluate the bending strength, i.e. modulus of rupture (MOR), of such finger-jointed samples, in particular when the acetylated wood was combined with unmodified wood, in this case, Norway spruce (Picea Abies L. Karst) (US), see example in Figure 1. The finger-jointing were performed at Moelven Töreboda by applying their existing industrial procedures. In total, five different of finger jointed sample groups were prepared combining the different specimens: APS-APS, AJP-AJP, US-US, APS-US, and AJP-US. Standardized procedures were used to determine the MOR of the finger-jointed samples, both unexposed at the factory condition state and after a water-soaking-drying cycle.

In addition, the experiments also included determination of the moisture content (MC), density, and modulus of elasticity (MOE) (in bending along the grain) of the individual specimens. At the unexposed state, the APS-APS samples showed the highest MOR of 63,1 MPa, while those of the AJP-AJP showed the lowest value of 42,4 MPa. The corresponding values for the US-US, AJP-US and APS-US samples was 56,7, 47,5 and 46,9 MPa, respectively. In contrast to a typical wood failure for the US-US samples, a low amount of wood failure was observed in all cases involving the acetylated wood, indicating a low adhesive anchoring in the wood substrate at the finger-joint, although a surprisingly high strength was obtained for the APS-APS samples. A significantly lower MC content of 4,9 % and a remarkably low value of 1,7 %, was found for the APS and AJP, respectively, compared with 9,2% for the US. The significantly lower MC combined with an assumed increased hydrophobicity of the acetylated wood possible causes a less effective MUF-wood bonding, or adhesion, compared with that of the unmodified wood. Possible, so-called over penetration of the MUF resin in the acetylated wood could also be an explanation for the poor wood-adhesive anchoring. The MOE of the individual APS, AJP and US specimens was 12,6, 8,3 and 11,4 GPa, respectively, indicating a significantly lower mechanical performance of AJP, and hence also of finger-joints of AJP, despite its very low MC, possible due to a higher microfibril angle in the cell walls in juvenile wood compared with mature wood. No clear correlation was found between the MOR and density of the acetylated samples. For the samples exposed to a water-soak-drying cycle, the highest MOR, and lowest reduction of 14 % compared with the unexposed state, was obtained for the US-US samples, whereas all samples involving the acetylated wood showed a distinctly higher reduction.

The MOR of the AJP-AJP and AJP-US samples were reduced with 47 % and 50 %, respectively, while the MOR of the APS-APS and APS-US samples were reduced with 43 % and 23 %, respectively. It should be emphasized, however, that after the standard drying-time, which was the same for all samples, the acetylated samples, compared with the untreated ones, did not dry out to the same level as for the dry unexposed state, i.e. the acetylated samples had a high MC of ca 30-40% in these MOR tests. This high MC level could be the main reason for the dramatic strength losses. Furthermore, a less efficient wood-MUF adhesion as well as the drying under acidic conditions may also be possible causes for the reduced bending strength of the finger-jointed samples with acetylated wood.

Figure 30. Finger-joint of (left) acetylated juvenile pine (AJP) and (right) untreated spruce (US).

7.2 Glulam hybrids of acetylated birch and untreated spruce

The aim of this work, see [2], was to investigate the possibility to use so called hybrids of chemically modified birch and untreated spruce, see Figure 2. The idea is to gain a product with a high resistance against biological degradation in the parts that are hard to protect, for example near the connections, without using modified wood for the entire beam. By finger jointing the two materials the acetylated part can be used near the connection and then the untreated part can start where it is easier to protect it with conventional wood protection methods.

More specific this work aimed to see how a beam of this sort behaves when tested in four-point bending. The goal was to see how and where the failure occurred and what bending strength and Young's modulus the beams had. Also, smaller tests were performed to see how the acetylation affected the material properties of the birch. The study is based on both experimental trials and numerical analysis. Four-point bending was performed on beams and smaller test pieces. A numerical analysis was performed to try to predict the failure behaviour and also to investigate how the finger joints are affected by the big differences in swelling behaviour between untreated and acetylated wood since the latter is more dimensionally stable.

The result indicated that the acetylated birch had a larger scatter in the material parameters compared to untreated birch. The tests performed on the beams showed that the failure always was initiated in the finger joint, either because of the glue between the parts or local defects in the wood fingers. The result also indicated that the beams obtained a higher stiffness when the mid-part of the beams consisted of birch. Also the finite element calculations indicated that the finger joints are the biggest problems and also that the swelling of spruce can lead to problems between the materials.

Figure 31. Center section of a glulam hybrid of acetylated birch (mid part) and untreated spruce (outer parts)

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7.3 Prediction of the tensile strength of birch plywood at varying angles to grain

In this study by Wang et al. (2020) birch (Betula pendula and Betula pubescens) is utilized as raw material, assigning high density, stiffness and strength to plywood. The tensile strength of birch plywood at varying angles (0-90 degree) to the face grain was predicted analytically by using one linear and two quadratic failure criteria. Conclusions drawn revealed that all the three failure criteria predict the lowest tensile strength at around 45 degree to the face grain. The linear failure criterion reveals lower results than the other two quadratic failure criteria where the empirical Norris and Tsai-Hill failure criteria show high similarities. Experimental tests will be carried out in the future to provide a database of such mechanical properties and determine the applicability of each failure criterion for birch plywood.

7.4 Super liquid-repellent acetylated birch

The aim of this work, see [3] and [4],) was to functionalize acetylated birch to obtain superhydrophobicity, i.e. acetylated wood surfaces with extreme liquid repellence of water. The motive for such a surface modification is to develop acetylated wood surfaces with a reduced risk of discoloration (mainly of mold) but also a reduced risk of deacetylation of the bulk of the wood. Veneer samples of birch (B) and acetylated birch (AB) were surface-modified (SM) by non-fluorinated silicone nanofilaments (SMB and SMAB), see the principle shown in Figure 3. Results showed contact angles greater than 160° towards water on both the SMB and the SMAB samples. In multicycle Wilhelmy plate tests, see example plots in Figure 4, the SMAB samples showed the lowest water uptake. In fact, a pronounced enhanced water resistance, i.e. a super-liquid repellence was observed for SMAB resulting in very low water uptake of 3 ±1 wt% after 100 cycles, which was about 29, 5 and 3 times lower than that of the birch, acetylated birch and surface-modified birch, respectively, see Figure 5. In addition, the SMB exhibited more color change than the SMAB, which was caused by the release of hydrochloric acid during the surface modification process.
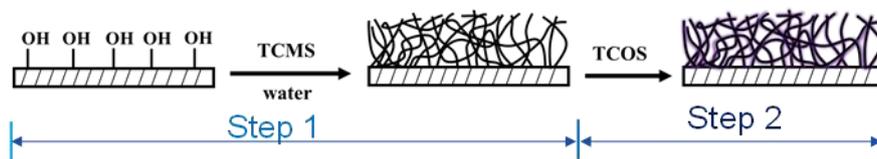
Figure 32. The principle of the applied surface modification concept based on a hydrophobized coating of silica nanofilaments.
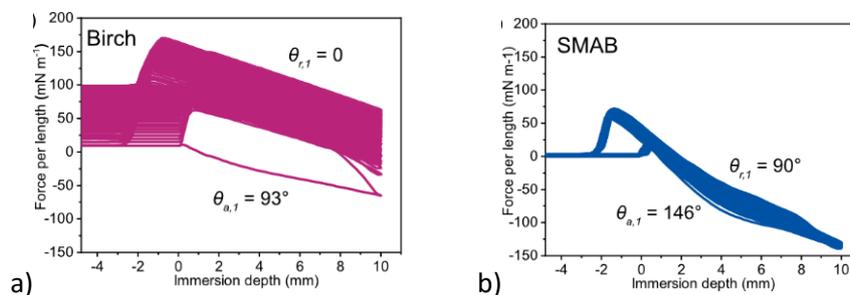

Figure 33. Force per perimeter length as a function of immersion depth sample position for 100-cycle Wilhelmy plate measurements for (a) birch, and (b) SMAB.
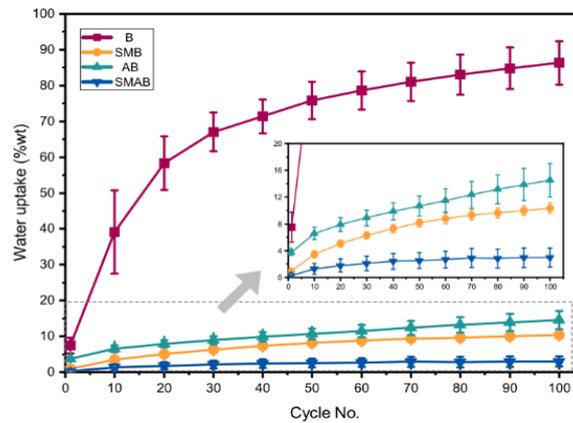
Figure 34. Water uptake with standard deviations as a function of cycle number for birch (B), acetylated birch (AB) and SMB and SMAB prepared at 5 h.

7.5   References

[1]   Wincrantz, C. (2018). Finger-jointing of acetylated Scots pine using a conventional MUF resin. Master Thesis. TRITA-ABE-MBT-18467. KTH Royal Institute of Technology, Stockholm, Sweden. http://urn.kb.se/resolve?urn=urn:nbn:se:kth:diva-240655

[2]   Sjölund, M. och Stenis N. (2019). Limträhybrider av acetylerad björk och obehandlad gran: En experimentell och numerisk analys av vissa mekaniska egenskaper och fuktbeteende. Master Thesis. TRITA-ABE-MBT-19534. KTH Royal Institute of Technology, Stockholm, Sweden. http://urn.kb.se/resolve?urn=urn:nbn:se:kth:diva-255272

[3]   Yin, H., Sedighi Moghaddam, M., Tuominen, M., Dėdinaitė, A., Wålinder, M., Swerin, A. (2020) Non-fluorine surface modification of acetylated wood for improved water repellence. Submitted manuscript.

[4]   Yin, H., (2020). Surface-modified wood based on silicone nanofilaments for improved liquid repellence. Licentiate Thesis in Civil and Architectural Engineering with specialization in Building Materials, KTH Royal Institute of Technology, Stockholm, Sweden.

[5]   Wang, T., Wang, Y., Crocetti, R., Wålinder, M. (2020). Prediction of the tensile strength of birch plywood at varying angles to grain. Oral presentation (first author). In: Sipi, M., Rikala, J. (Eds) Proceedings of the 16th Annual Meeting of the Northern European Network for Wood Science and Engineering – WSE2020, 1–2 December, Helsinki, Finland, p 80-82.

8 Dowel-type joints and glued-in rods

8.1 Dowel type joints with acetylated pine

8.1.1 Background and overview of tests performed

The brittleness of the wood material is an important factor to consider in the design of dowel type joints. In contemporary structural timber codes, design provisions typically specify rules for placement of fasteners and dowels, which is a means to implicitly take the brittleness into account.

By prescribing end- and edge distances and the distance between fasteners in a joint, it is assumed that brittle failure modes can be avoided. In other cases, e.g. as the design formula for tension at angle to the grain for a single fastener, Eurocode 5 includes in the design equation a model parameter which is based on an assumption of the fracture energy of the material. Thus, even though fracture energy is not explicitly stated as a material parameter in the codes, it is indeed an important parameter to consider.

In order to study how the acetylation affects the brittleness of dowel-type connections, timber members of both unmodified and acetylated Scots pine (*Pinus sylvestris*) were examined.

Dowel-type connections were tested by loading perpendicular and parallel to the grain. The specimens and the configuration of all tests were designed such as to provoke brittle failures. The two test set-ups used in this study are shown in Figure 35. In addition to the dowel joint tests, also embedment strength was tested, for further details, see [1].
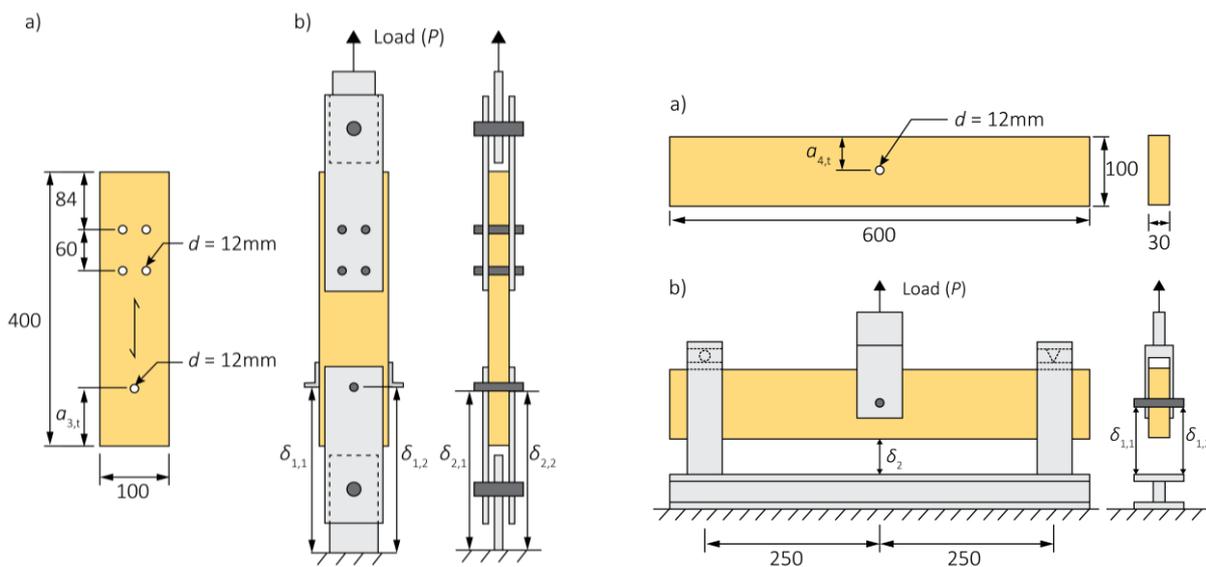
Figure 35. Test set-ups used in testing of dowel-type joints. Left: loading along grain tests. Right: loading perpendicular to grain tests (splitting).

8.1.2 Results and conclusions

Based on findings from this study, the following can be concluded:

The acetylated wood was found to have a significantly higher embedment strength parallel to the grain. No such increase was found for the embedment strength perpendicular to the grain. The

increased embedment strength parallel to the grain was not well predicted by using the measured density to estimate it according to EC5. The prediction gave a large underestimation of embedment strength along grain (-24%) and a less severe underestimation perpendicular to grain (-11%).

The increased embedment strength of the acetylated wood lead to an increased load-bearing capacity of the joint, compared to unmodified wood. This capacity was conservatively estimated by applying the EC5-design approach, but the acetylated wood specimens did show premature brittle failure modes. Thus in order to obtain a ductile response, the use of reinforcement was recommended. If the capacity level is less of a concern, a lower steel grade could be used instead.

In the test of joints loaded perpendicular to the grain, the acetylated wood resulted in lower load bearing capacity, compared to the unmodified wood. In any case, the connections failed due to splitting, with a crack propagating along the grain. The load bearing capacity was underestimated by the EC5-formulae in most cases, and for all cases involving the acetylated wood. Thus it was recommended that the design provisions should be adjusted accordingly.

It was finally concluded that there is a need to further investigate the brittleness of dowel-type connections made from acetylated wood, including also multiple dowel connections, influence of moisture and duration-of-load effects.

8.2   Glued-in rods with acetylated birch

8.2.1   Examples of application of glued-in rods on outdoor structures

The below pictures (Figure 36Figure 37) show examples of glued-in rods used in practice. The main advantages of the glued-in rod connection is that a strong, stiff and aesthetically appealing connection can be achieved. Since the glued-in steel parts are inert in terms of moisture induced deformations, moisture variations and especially outdoor structures constitute a special challenge. Consequently, it is of considerable interest to investigate the possibility of using acetylated wood in this type of connection, since the acetylated wood is much more dimensionally stable than untreated wood.
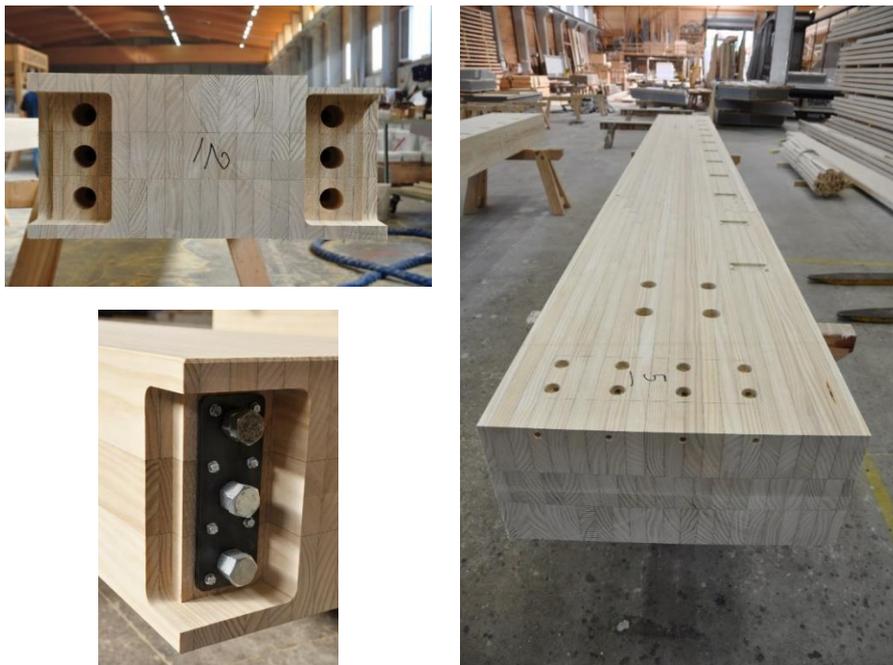

Figure 36. Manufacturing of glued-in rod connections at partner Simonin.

Figure 37. Examples of use in outdoor structures. Left: Portal frames with post/beam moment resistant connections. Right: Rafters with moment-resistant connections fixed on a wall.

8.2.2 Material and methods

Solid Birch, both control and acetylated, was sent to partner Simonin in the form of planks, Figure 38. The planks were glued together with a MUF adhesive to obtain specimens of dimensions suitable for testing of glued-in rods. From the raw material, specimens with dimensions 80×80×714 mm were manufactured, made of untreated (control) and acetylated birch. Steel rods of 16 mm diameter, metric thread M16, were glued with an epoxy resin according to the provisions of the Resix® connection system (licence Simonin SAS France), see Figure 38.

Figure 38. Solid birch (left) was used to manufacture specimens for testing of glued-in rods (right).

Testing of the samples was performed by partner Simonin, making use of their equipment used in internal testing. The tests were performed as pull-pull tests, and the set-up used is shown in Figure 39

Figure 39. Internal testing at Simonin SAS (France).

8.2.3 Results

The test results are summarised in Table 11. For this type of connection with glued-in rods, brittle failure is always observed as long as the failure is not by yielding of the rod. In the tests reported here, the observed failure modes mostly conformed to the usual failure mode for other wood species, see Figure 40. Of special interest is that the failure modes for untreated and acetylated Birch are also quite similar. The capacity of the connection is lowered by approximately 40% for the acetylated birch samples compared to the untreated birch samples. Nevertheless, the pull-out resistance of the acetylated wood remains sufficiently high to be an efficient way of connection, especially for outdoor structures.

Table 11. Test results from pull-out tests of glued-in rods in Birch. Tests performed at Simonin SAS.

	Material	Humidity (%)	Capacity (kN)	Shear stress (MPa)
Cross section: 80×80 mm² Rod: φ16 mm, M16 Glued length: 200 mm	Acetylated Birch	4.9	76.6	7.6
		3.9	79.4	7.9
		4.3	77.0	7.7
		4.5	74.8	7.4
		4.3	81.1	8.1
	Mean	*4.4*	*77.8*	*7.7*
	Std. dev.	*0.36*	*2.48*	*0.25*
	Untreated Birch (control samples)	7.5	118.6	11.8
		8.9	131.2	13.1
		9.4	135.3	13.5
		8.4	130.7	13.0
		7.7	136.2	13.6
	Mean	*8.4*	*130.4*	*13.0*
	Std. dev.	*0.80*	*7.05*	*0.70*

Figure 40. Failure mode for acetylated Birch samples.

8.3 References

[1] Forsman, K., *Fracture behaviour of acetylated wood – Material characterisation and dowel-type connections*. Licentiate dissertation, TVSM-3081, Structural Mechanics, Lund University.

9   Conclusions and further studies

Some of the main overall conclusions from the project are:

- There is a technical potential in using acetylated wood for outdoor load bearing structures
- The acetylation processes used in today's commercial process for non-structural material is probably possible to use for sapwood of Scots pine with only minor changes.
- The challenge of using Scots pine instead of radiate pine is related to the detection of heartwood and selection of permeable raw material.
- Acetylation of solid birch is challenging, and if this is aimed for, substantial development and calibration of especially the drying sequence of the process is needed.
- Acetylation of birch veneers is possible, although severe cracking can occur.
- Acetylated wood is more demanding to glue and acetylated birch (hardwood) is more difficult than acetylated pine (softwood). From what is known for the time being the choice of available adhesive systems is rather limited. Some commercially available adhesive systems could be used but would need further development of the gluing process (time/temperature/pressure).
- As regards wetting, acetylated wood behaves differently as compared to unmodified wood. Although acetylated birch is not considerably more hydrophobic than unmodified birch, it is significantly less prone to water uptake when exposed to a number of wetting cycles. Results indicate that PRF is the most robust adhesive and has the best gluing performance on both acetylated pine and birch.
- The influence of acetylation on the fracture energy of the wood is substantial, 35-50% decrease depending on relative humidity level.
- The decrease in fracture energy is to a large degree, but not solely, explained by the lower equilibrium moisture content of acetylated wood as compared to untreated wood.
- The design provisions of e.g. EC5 need most probably to be adapted for design situations where the fracture energy of the structure is of importance (note that this influence is currently *not* explicitly stated).

As regards further studies, it is recommended to investigate:

- Possible improvements in gluing process parameters.
- Adaption of acetylation process for other species and formats (birch, veneers).
- Development of robust process for selection of permeable Scots pine for acetylation.
- Further studies on mechanical behaviour to include duration of load effects, which might be less pronounced for acetylated wood as compared to untreated wood.

10 Research output

Journals

Forsman, K., Serrano, E., Danielsson, H., Engqvist, J. Fracture characteristics of acetylated young Scots pine*. European Journal of Wood and Wood Products volume 78, pp. 693–703(2020)*

Forsman, K., Fredriksson, M., Serrano, E., Danielsson, H. Moisture-dependency of the fracture energy of wood: A comparison of unmodified and acetylated Scots pine and birch. Accepted for publication in *Holzforschung*.

Conferences

Forsman, K., Danielsson, H. and Serrano, E. Fracture Characteristics of Acetylated Scots Pine - An experimental and numerical investigation. CompWood 2019, 17 – 19 June 2019, Växjö, Sweden.

Forsman, K., Serrano, E. and Danielsson, H. Modified wood in outdoor load-bearing structures. Oral and poster presentation. Young researcher's program of the 2019 Marcus Wallenberg Prize events, 7-8 October, 2019.

Forsman, K., Serrano, E. and Danielsson, H., Fracture characteristics of acetylated scots pine and birch, Oral presentation at 15th Annual Meeting of the Northern European Network for Wood Science and Engineering, Lund, 9-10 October 2019.

Yin, H., Sedighi Moghaddam, M., Tuominen, M., Dėdinaitė, A., Wålinder, M., Swerin, A. (2020) Non-fluorine surface modification of acetylated wood for improved water repellence. *Submitted manuscript*.

Forsman, K., Fredriksson, M., Serrano, E. Danielsson, H. Moisture-dependency of the brittleness of acetylated wood. Oral presentation at 16th Annual Meeting of the Northern European Network for Wood Science and Engineering, Helsinki, 1-2 December 2020 (online conference).

Wang, T., Wang, Y., Crocetti, R., Wålinder, M. (2020). Prediction of the tensile strength of birch plywood at varying angles to grain. Oral presentation (first author). In: Sipi, M., Rikala, J. (Eds) Proceedings of the 16th Annual Meeting of the Northern European Network for Wood Science and Engineering – WSE2020, 1–2 December, Helsinki, Finland, p 80-82.

Licentiate dissertations

Forsman, K., *Fracture behaviour of acetylated wood – Material characterisation and dowel-type connections*. Licentiate dissertation, TVSM-3081, Structural Mechanics, Lund University.

Yin, H. (2020). Surface-modified wood based on silicone nanofilaments for improved liquid repellence. Licentiate Thesis in Civil and Architectural Engineering with specialization in Building Materials, ISBN: 978-91-7873-692-8, TRITA-ABE-DLT-2038, KTH Royal Institute of Technology, Stockholm, Sweden. http://urn.kb.se/resolve?urn=urn:nbn:se:kth:diva-285601

Manuscripts

Forsman, K., Serrano, E., Danielsson, H. Brittleness of dowel-type connections made from acetylated wood. To be submitted.

Master's dissertations

Wincrantz C. Finger-jointing of acetylated Scots pine using a conventional MUF resin. (2018). (TRITA-ABE-MBT). Available from: http://urn.kb.se/resolve?urn=urn:nbn:se:kth:diva-240655

Sjölund, M., & Stenis, N. (2019). Limträhybrider av acetylerad björk och obehandlad gran : En experimentell och numerisk analys av visa mekaniska egenskaper och fuktbeteende (Dissertation). Retrieved from http://urn.kb.se/resolve?urn=urn:nbn:se:kth:diva-255272

Lai, G. and Plönning, S. (2019) Fracture characteristics of acetylated birch - Experimental and numerical studies, Master's thesis, Report TVSM-5234, Division of Structural Mechanics, Lund University, Lund Sweden. http://lup.lub.lu.se/student-papers/record/8990739