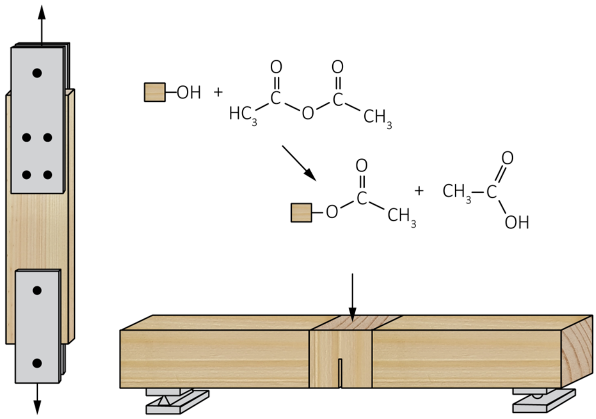

FRACTURE BEHAVIOUR OF ACETYLATED WOOD
Material characterisation and dowel-type connections

KARIN FORSMAN

Structural Mechanics

Licentiate Dissertation

DEPARTMENT OF CONSTRUCTION SCIENCES

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LICENTIATE DISSERTATION

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Finally, without my family, my friends, and my partner Vedad Alic, I would have lost my mind a long time ago – thank you for reminding me of what really matters in life.

Lund, December 2020
Karin Forsman

Abstract

In order to increase the competitiveness of timber as a building material in outdoor applications, durability and dimensional stability must be ensured. Acetylation enables an environmentally friendly way to increase both durability and dimensional stability of wood, without introducing harmful substances to the environment. The focus of the present research is to examine the possibility of adding a structural value by acetylation of wood species native to the Nordic region, rarely used for load-bearing structures outdoors due to poor durability and dimensional stability. Yet, before a large scale use is possible, the mechanical properties of acetylated wood must be examined. The research presented in this dissertation focuses on the brittleness of acetylated wood, both at a clear wood level as well as in structural applications.

Fracture characteristics are hereby defined by properties that influence the brittleness, considered by the stiffness, tensile strength and fracture energy. For Scots pine conditioned at a relative humidity of 60% and a temperature of 20°C, no significant impact of the acetylation process was found for the stiffness along the grain, nor for the tensile strength perpendicular to the grain. However, for Scots pine and birch examined at various relative humidity levels, the fracture energy was found to be significantly reduced for acetylated wood at relative humidity levels up to 97%. The largest difference between unmodified and acetylated wood of the same species and at equal climate conditions was approximately 50%. The studies demonstrated a clear moisture-dependency of the fracture energy for both unmodified and acetylated wood, but it was suggested that the fracture energy is lower for acetylated wood compared to unmodified wood at similar moisture contents. The lower fracture energy of acetylated wood when compared to unmodified wood at equal relative humidity levels can thus partly, but not solely, be explained by the reduced hygroscopicity of acetylated wood.

To evaluate the implications of the increased brittleness in terms of structural applications, single dowel-type connections made from acetylated and unmodified Scots pine were studied. Results were compared to Eurocode 5 estimations to evaluate the validity of current design provisions for acetylated wood. It was found that, for all the tested end-distances for loading parallel to the grain, joints made from acetylated wood failed in a brittle manner. Nevertheless, connections made from acetylated wood demonstrated a significantly higher embedment strength and load-bearing capacity parallel to the grain, and Eurocode 5 provided conservative estimations. For loading perpendicular to the grain, a reduced splitting capacity was found for acetylated wood compared to unmodified wood, and here the load-bearing capacity was overestimated by Eurocode 5.

The most important conclusion from the research presented herein is the increased brittleness of acetylated wood compared to unmodified wood. Special attention is, hence, required in structural design using acetylated Scots pine and birch. In case of dowel-type connections, or other loading situations where stress concentrations occur, measures should be taken to

avoid premature brittle failure modes. This risk may for instance be limited by increasing spacings between fasteners as well as end- and edge-distances, and/or reinforcement of joints. Further studies are needed to increase the knowledge of how the acetylation process will impact load-bearing structures. Although the research presented herein reveals one disadvantage with acetylated wood, it can still outperform unmodified wood in moist conditions thanks to e.g. increased dimensional stability and durability.

Contents

Paper A
Fracture characteristics of acetylated young Scots pine.
Karin Forsman, Erik Serrano, Henrik Danielsson, and Jonas Engqvist.
European Journal of Wood and Wood Products 78, 693-703(2020).
Contributions
K. Forsman: Main author of the paper. Planning, designing, and conducting experimental work. Processing and interpretation of results.
E. Serrano: Stipulated the research question. Contributing by discussions and comments on methods, interpretation of results, and manuscript.
H. Danielsson: Contributing by discussions and comments on methods, interpretation of results, and manuscript.
J. Engqvist: Contributing by experimental set-ups and through discussions and comments on experimental methods, interpretation of results, and manuscript.

Paper B
Moisture-dependency of the fracture energy of wood: A comparison of unmodified and acetylated Scots pine and birch.
Karin Forsman, Maria Fredriksson, Erik Serrano, and Henrik Danielsson.
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Contributions
K. Forsman: Main author of the paper. Planning, designing, and conducting experimental work. Processing and interpretation of results.
M. Fredriksson: Designing and conducting experimental work on sorption isotherms. Contributing by discussions and comments on methods, interpretation of results, and manuscript.
E. Serrano and H. Danielsson: Contributing by discussions and comments on methods, interpretation of results, and manuscript.

Paper C
Brittleness of dowel-type connections made from acetylated wood.
Karin Forsman, Erik Serrano, and Henrik Danielsson
Manuscript to be submitted.
Contributions
K. Forsman: Main author of the paper. Planning, designing, and conducting experimental work. Processing and interpretation of results.
E. Serrano and H. Danielsson: Contributing by discussions and comments on methods, interpretation of results, and manuscript.

Part I

Introduction and overview

1 Introduction

1.1 GENERAL BACKGROUND

Timber is one of the oldest building materials, providing several benefits such as a high strength to weight ratio and insulation properties superior to many other structural materials. Wood is also easy to process, hence, it opens possibilities for new architectural expressions, and is often perceived as aesthetically appealing. Still, one of the most recognized virtues of wood is its environmental benefits, being a renewable material. During the lifetime of a tree, it absorbs carbon dioxide from the atmosphere, which is stored in the tree after being harvested. In that sense, buildings made from timber serve as carbon dioxide storages during their lifetime.

Wood clearly provides numerous possibilities, yet, some inevitable drawbacks due to its biological nature must be recognized when used in moist conditions. Wood is a hygroscopic material, meaning that it absorbs water from the surrounding atmosphere. When exposed to changes in moisture, the material will swell and shrink, which may lead to crack formation and propagation. Moreover, many wood species suffer from low durability when exposed to high moisture contents. Without any protection of the timber, the long-term presence of moisture will lead to degradation of the wood substance.

For a long time different kinds of preservatives have been used to prevent biological decay. However, several preservatives contain toxic substances, harmful to the environment. To improve both durability and dimensional stability, without the use of hazardous substances, alternative wood modification methods have been developed. By changing the chemistry of the wood, it has been acknowledged that it is possible to alter its properties – reducing its hygroscopicity. Wood modification methods are often divided into thermal and chemical methods. Thermal modification methods are based on subjecting the wood to high temperatures in absence of oxygen. The heating process results in changes within the chemical structure of the wood substrate, reducing its hygroscopic properties. Chemical modification methods are based on the reaction between a chemical reagent and the wood polymers, forming stable bounds. Among chemical modification methods, acetylation is one of the most studied. By occupying free hydroxyls, that normally bind water molecules, it enables an environmentally friendly way to increase both durability and dimensional stability of numerous wood species. Acetylated wood is commercially available, but main areas of use today are for non-structural applications.

1.2 PROBLEM STATEMENT

Chemical modification methods open up new possibilities, where properties of wood species with low durability and dimensional stability can be improved and, thus, gain new areas of use. However, altering the chemical structure of the wood substance will also affect its mechanical properties. Several studies on various wood species have been conducted with regard to the effects of acetylation on basic mechanical properties. Meanwhile, there is a lack of research performed on the impact of acetylation on fracture characteristics of wood. Reiterer and Sinn [1] presented a study indicating a reduced fracture energy of 20% for acetylated Spruce. Fracture characteristics are decisive in several structural applications, since the occurrence of stress concentrations may lead to crack propagation, with possible catastrophic outcomes.

The reasons for the increased brittleness of acetylated wood compared to unmodified wood are unknown. Since previous studies have only considered fracture characteristics of unmodified and acetylated wood conditioned at equal relative humidity levels, samples have so far only been compared at unequal moisture contents. It may thus be hypothesised that the increased brittleness is simply a consequence of a drier, hence, more brittle material.

Further, to enable the use of acetylated wood in structural applications, the impact of the altered properties need to be evaluated for structural applications. A previous study [2] indicated an increased brittleness of dowel-type connections made from acetylated wood. Design criteria to prevent premature brittle failures included in Eurocode 5 are implicitly based on fracture characteristics. Thus, the validity of such design criteria, e.g. in terms of minimum end- and edge-distances, may be questioned for a material with an increased brittleness.

1.3 AIM AND OBJECTIVES

1.3.1 Aim

The aim of the research has been to investigate the possibility of using acetylated young Scots pine and birch for load-bearing applications. Unlike commercially available acetylated wood, consisting of Radiata pine, the work presented here concerns wood species native to the Nordic region. These wood materials are rarely used for structural purposes in outdoor conditions, due to limited durability and dimensional stability. To increase durability, preservative treatments can be used, although the dimensional stability remains an issue. By acetylation, it is possible to increase both durability and dimensional stability, and limit today's use of toxic preservatives. If this can be done, without compromising the structural integrity, a structural value will be added to domestic forestry.

Before the use of acetylated wood in outdoor load-bearing structures can be realized on a large scale, knowledge of the effects of acetylation on a clear wood level of the material is essential. One aim of the research presented in this dissertation has been to investigate the impact of the acetylation process on the *fracture characteristics* of the wood material. The *moisture-dependency of the fracture energy* has also been studied, to gain knowledge about the impact for moisture conditions relevant in the design of load-bearing structures. Finally, the research has also investigated if *design provisions* must be revised to also include acetylated wood.

1.3.2 Objectives

The objectives of the research presented in this dissertation has been to study the effects of the acetylation process on:

- Fracture characteristics of young Scots pine, herein defined by the tensile strength perpendicular to the grain, stiffness parallel to the grain and fracture energy in tension perpendicular to the grain.
- Moisture-dependency of the fracture energy in tension perpendicular to the grain for young Scots pine and birch, including absorption isotherms at a temperature of 20°C.
- The load-bearing capacity and the embedment strength of dowel-type connections made from young Scots pine, loaded perpendicular and parallel to the grain.

1.4 RESEARCH APPROACH AND LIMITATIONS

The work has primarily comprised experimental studies to characterise the acetylated material in terms of fracture properties as well as structural properties of dowel-type connections. The aim has been to gain knowledge and input data for models to be used for numerical analysis of structural elements and joints made from acetylated wood.

Due to a restricted amount of acetylated wood material available, research has been focused on gaining insight and understanding on how properties are affected, rather than trying to verify design values. In the characterisation of a material, as well as development of design provisions, several samples must be studied to supply a statistical data base. Thus, it is stressed that the results presented herein, as well as in the published papers, should be viewed as phenomenological, rather than providing accurate quantification of material properties or joint capacities.

The research on fracture characteristics has been performed for small, clear wood samples. Thus, test samples are not representative for structural-sized timber, where natural imperfections are present. The justification of clear wood samples is, however, considered to be valid in the comparison of unmodified and acetylated wood.

The present research is further subjected to the following limitations:

- Only short-term loading is considered, i.e. long-term effects are neglected.
- Various relative humidity levels have been considered when determining the fracture energy, but for the stiffness, strength and load-bearing capacity of dowel connections, only one relative humidity level was regarded.
- The impact of temperature on examined characteristics has not been studied.

1.5 OUTLINE OF DISSERTATION

The dissertation consists of a general introduction, followed by a more detailed background to introduce the reader with less pre-knowledge within the research area. Thereafter, an overview of the research contributions is presented, where main findings from the appended papers are highlighted. Finally, concluding remarks and thoughts on further work are presented. In the following, each chapter is briefly introduced.

Chapter 1: Introduction

This chapter aims at introducing the reader to a general background. The research gap, from which the research of this dissertation has sprung, is defined. Moreover, aim and objectives are presented, as well as the research approach and limitations of the work.

Chapter 2: Background

Wood is a natural material, and to understand how it behaves mechanically, an understanding of its structure is essential. This chapter introduces the reader to a basic understanding of the microscopic, macroscopic and ultra-structure levels of wood, and how it is affected by moisture. Moreover, basic mechanical properties of wood are introduced, focusing on stiffness and strength. As the research considers acetylated wood, the process of acetylation is described more in depth, as well as how it affects physical and mechanical properties. A short introduction to the fracture behaviour of wood is presented. Moving forward from a material level to a structural level, the reader is introduced to how brittleness is regarded in Eurocode 5 (design provisions) – with a focus on dowel-type connections.

Chapter 3: Overview of present work

In this chapter, the work presented in papers A–C is briefly reviewed, including important aspects of test conditions as well as compiled versions of main findings. The chapter is divided into two sections, where the first is focused on characterisation of wood at a clear wood level, and the second on structural applications, i.e. dowel-type connections.

Chapter 4: Concluding remarks and further work

Herein, the main conclusions from the research performed within the scope of this dissertation are presented. Suggestions on further work are suggested.

2 Background

2.1 STRUCTURE AND PROPERTIES OF WOOD

> Good timber does not grow with ease;
> the stronger the wind, the stronger the trees
>
> Douglas Malloch

Wood is a natural composite, designed to supply the needs of the living tree, i.e. to transport water and nutrients, as well as to support rigidity for the tree to sustain wind-loads and self-weight. The optimization of the tree to these conditions results in different mechanical characteristics in different directions, with respect to the direction of the tree stem. To understand the mechanics of wood, and to be able to optimally exploit its potential, it is necessary to understand how it is structured – from the ultra-structure to a macroscopic level. This section aims at introducing the reader to a basic knowledge of the structure of wood, how it interacts with moisture, and how it behaves mechanically. The section is based on [3–7].

2.1.1 Wood anatomy

Softwood and hardwood

Wood is often classified as *softwood* or *hardwood*, botanically distinguished by the way they reproduce. In general, softwood comes from coniferous trees, where the term conifer translates to cone-bearer. Many conifers are evergreens, i.e. they stay green all year round, not shedding their leaves/needles. Softwoods are known as a gymnosperm as they reproduce by forming cones, containing uncovered seeds to be spread by the wind. Examples of softwoods include pines, cedars, spruces and firs. Hardwoods are often deciduous trees, i.e. they shed their leaves each fall. They are known as angiosperms, and their seeds contain some kind of covering. Hardwoods include species such as oaks, beeches, maples and birches. Hardwoods are in general, but not always, more dense, thus, harder compared to softwoods. Differences can also be found on a macroscopic and a microscopic level, which is covered in the following.

Macroscopic structure

The purpose of the tree stem is mainly to support and supply water and nutrients to the tree. A schematic illustration of a cross section of a tree stem is shown in Figure 2.1, defining parts of the macroscopic structure of wood, i.e. elements visible to the naked eye. The *bark* consists of two parts: A protective outer layer, and an inner layer responsible for transportation of nutrients produced in the crown during photosynthesis to the *cambium* and storage cells. The cambium is an extremely thin layer located inside of the bark, where new wood is formed by cell division. Wood (xylem) forms in the inner part of the cambium, whereas bark (phloem) is formed in the outer part.

Sapwood consists of wood cells serving as a storage recipient for water and energy reserve material, such as starch. The sapwood is responsible for transportation of water from the roots to the crown. For sawn wood, the sapwood is often characterised by poor durability. As old sapwood in the living tree loses its vitality in transporting water and storing nutrients, it undergoes heartwood formation, i.e. the cells close up. *Heartwood* contains inactive cellular tissue and serves as stabilising and strengthening of the tree. In many wood species, the accumulation of extractives in the heartwood gives it a comparably darker colour, as indicated in Figure 2.1. Heartwood is characterised by a lower porosity compared to sapwood. It is less permeable and is often more resistant to decay. The *pith* is located in the middle of the stem. Although most water and nutrients are transported vertically along the tree stem, some transportation also occurs across the tree, a process in which the *ray* cells are involved. Rays also act as storage areas.

Mesoscopic structure

Under temperate conditions, distinct growth layers appear, known as *annual rings*, or *growth rings*. These represent the climatic conditions under which the tree has grown, also depend-

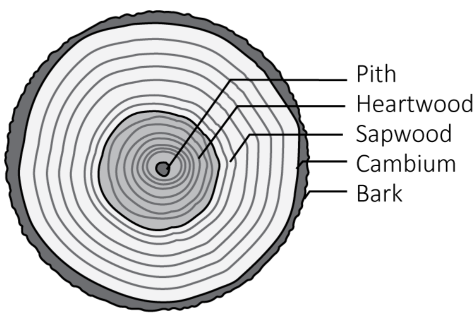


Figure 2.1: A schematic illustration of the cross section of a tree stem.

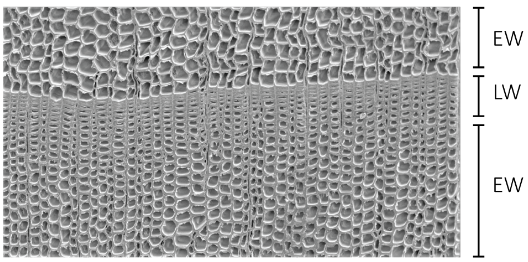


Figure 2.2: Scanning electron microscope image of spruce, illustrating earlywood (EW) and latewood (LW). Photo: Maria Fredriksson.

ing on species and silviculture. One year's wood formation is characterised by *earlywood* and *latewood* (Figure 2.2). Earlywood is formed during spring, when the tree is growing more rapidly, and the need for water supply is large. It consists of large pore volumes and thin-walled cells. Latewood is formed during late summer/autumn, when the water supply loses vitality, and the tree grows more slowly. The formation of cells can be focused on strength, and it is characterised by thicker cell walls. The difference in thickness is often visible from colour changes, although this difference is more pronounced for softwoods compared to hardwoods. The density and strength of the wood will depend on the width of the annual rings, and the proportion of latewood versus earlywood. The innermost (about 15–25) growth rings consist of *juvenile wood*, formed during the first decades of growth of the tree. The juvenile wood is known to have a much lower density and longitudinal stiffness than the surrounding material.

Microscopic structure

Softwood and hardwood differ in terms of microscopic structure. Softwoods have a simpler structure and are more homogeneous compared to hardwoods. The wood structure comprises two cell types: *tracheids* and *parenchyma*. Tracheids in softwoods are responsible for both water transportation and support, and are arranged in the longitudinal direction of the tree. For softwoods, cell walls of the tracheids are aligned in the radial but not in the tangential direction. Evolutionary, hardwoods developed later than softwoods. A scanning electron microscope (SEM) image of birch is shown in Figure 2.3, illustrating the more complex structure of hardwoods (c.f. Figure 2.2 for softwood). Hardwoods consist of specific cell types for different functions; instead of tracheids, *vessels* are responsible for water transportation and *fibres* for providing support of the tree. Common to both is that the main cell type in the wood (tracheid/fibre) serves some supporting function, giving mechanical strength to the tree. The tracheids or fibres (henceforth referred to as fibres) are formed as hollow tubes. Water transportation within the cell structure occurs within the *lumen* (i.e. the void at the centre of the cell), and to adjacent cells through systems of pits.

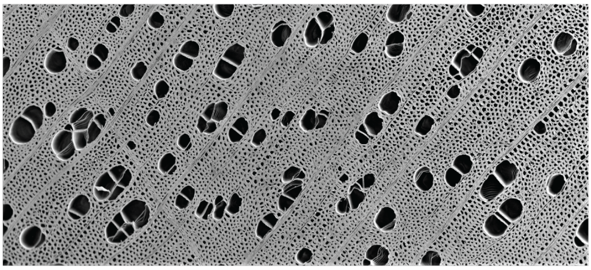


Figure 2.3: Scanning electron microscope image of birch, a diffuse-porous wood. Photo: Maria Fredriksson.

Ultra-structure

The cell walls are mainly composed of cellulose, hemicellulose, and lignin. Together, they form microfibrils that compose the cell wall. The microfibrils can be viewed as thread-like collections of cellulose molecules with high tensile strength, contained in the matrix material lignin, with hemicelluloses (smaller and branched molecules) acting as linkage between the lignin and the celluloses. The angle between the main direction of the fibre and the microfibril defines the *microfibril angle*.

A schematic illustration of the cell wall is shown in Figure 2.4, where it is divided into three main regions: the *middle lamella*, the *primary wall* and the *secondary wall*. The middle lamella acts as an adhesive between different cells, and is mostly composed of lignin. The primary wall is an extremly thin layer, with varying microfibril angles. The term compound middle lamella is sometimes used to describe the primary wall and the middle lamella together. The secondary wall that surrounds the lumen can be divided into three sub-layers: the outer layer (S1), the

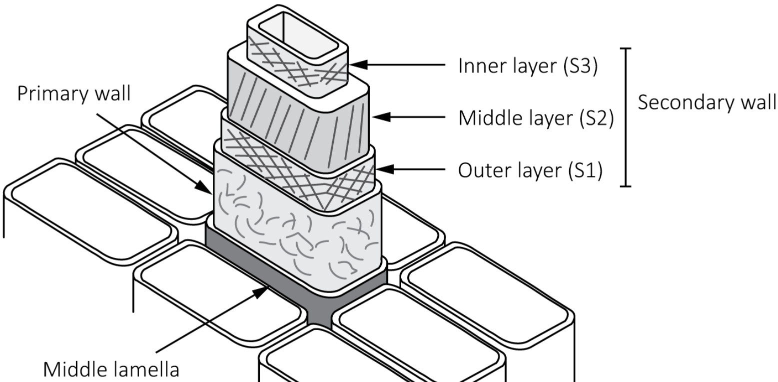


Figure 2.4: Schematic illustration of the cell wall at an ultra-structure level.

middle layer (S2) and the inner layer (S3). The S1 and the S3 layers are thin, with relatively large microfibril angles. Contrary, the S2-layer is the thickest layer and is characterised by low microfibril angles. Due to the comparably large thickness and longitudinally orientated microfibrils, the S2 layer will to a large extent dictate the structural properties of the material.

2.1.2 Wood-moisture interaction

Given the nature of wood, with it cells being responsible to supply the tree with water, it is not surprising that it contains and interacts with water even after processing. The moisture content of wood u (often expressed in percent) represents the ratio between the weight of the water contained in the wood, divided by the oven-dry weight of the wood substance:

$$u = \frac{m - m_{dry}}{m_{dry}} \tag{2.1}$$

where m is the weight of the wood substance and the contained water, and m_{dry} the weight of the dry wood substance. Water exists within the cell walls (molecularly bounded water) and as free water within cell cavities (capillary water). The moisture content of wood in its green state is approximately 60% to 200%, depending on the season of the year and location within the wood substance. The free water separates faster than the bound water, down to a moisture content of approximately 27%–30%. Above this range, a change in moisture content is known not to significantly affect strength or dimensions of the wood. The theoretical stage at which all free water is removed is referred to as the fibre saturation point (FSP). In practice, a well defined transition does not occur, thus, the FSP should rather be considered by a range of moisture contents.

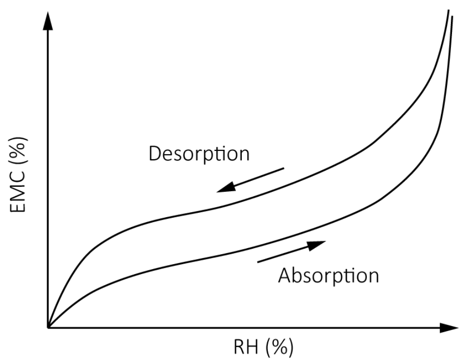


Figure 2.5: Absorption and desorption isotherms, describing the relation between the relative humidity (RH) and the equilibrium moisture content (EMC) at a given temperature.

The interaction between wood and moisture is affected by both temperature and relative humidity. When wood is in equilibrium with the ambient temperature and relative humidity, the moisture content is referred to as the equilibrium moisture content (EMC). The relation between relative humidity and EMC is described by sorption isotherms, illustrated in Figure 2.5. Two separate isotherms are used to describe the two processes of increasing (absorption) and decreasing (desorption) EMC. The discrepancy between the isotherms is known as hysteresis, and the ratio between the two EMC:s at a given relative humidity typically varies from 0.8–0.9 depending on species and temperature.

Most mechanical properties are known to depend on the cell wall moisture content, rather than the total moisture content. The range where the cell wall water uptake is dominant is referred to as the hygroscopic range. The water is bound to hydroxyls within the cell wall, where hemicellulose has the highest sorptive capacity, followed by cellulose and lignin. When water is removed from the cell wall, the distance between the microfibrils is reduced, which results in increased interfibrillar bonding, which in turn, increases strength and stiffness of the wood. Addition or removal of water molecules to the cell walls will also make the wood swell or shrink, resulting in issues with dimensional stability of the material. Due to non-uniform swelling and shrinkage, and/or due to restraining effects from adjacent materials or supporting conditions, cracks may form and propagate. Depending on the considered direction of the wood, the swelling/shrinking will vary. For example, the moisture induced strain is 5–10 times larger perpendicular to the grain compared to parallel to the grain.

2.1.3 Strength and stiffness properties

Wood is a strongly heterogeneous material, where the microscopic structure affects how the material behaves mechanically. For simplicity, wood can be described at a macroscopic level as a cylindrical orthotropic material, defined by the longitudinal (L), tangential (T) and radial (R) directions, illustrated at both a microscopic and a macroscopic level in Figure 2.6. Instead of applying a cylindrical coordinate system, a rectilinear coordinate system is sometimes used as a simplification. Such an approach then neglects the influence of the annual ring curvature. This can be justified for samples of small dimensions, located at a distance from the pith.

Stiffness properties

The linear-elastic relationship between stresses and strains can be assumed to follow Hooke's generalized law. This relationship describes a linear relation between the stress and strain components. Considering wood as a 3D continuum, assuming small strains, the linear elastic behaviour is given by:

$$\boldsymbol{\varepsilon} = \mathbf{C}\boldsymbol{\sigma} \tag{2.2}$$

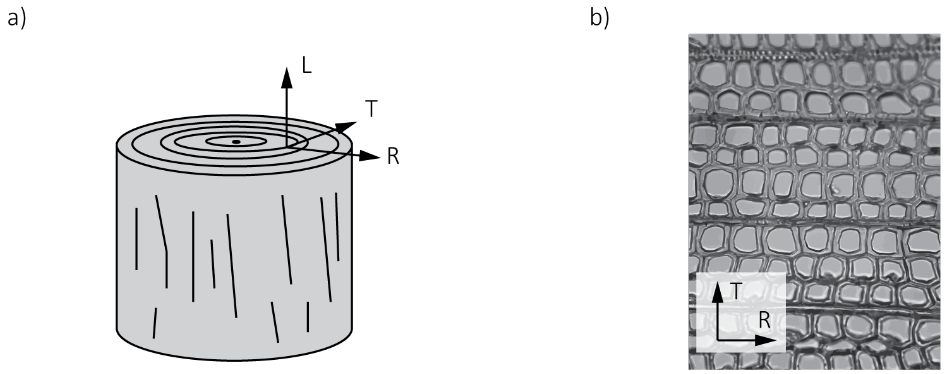


Figure 2.6: Definitions of the longitudinal (L), radial (R) and tangential (T) directions of wood, defined at a macroscopic (a) as well as a microscopic level (b). Photo: Maria Fredriksson.

where $\boldsymbol{\varepsilon}$ is the elastic strain tensor and $\boldsymbol{\sigma}$ the stress tensor. With principal directions defined along the material axes LRT, the strain and stress tensor are commonly organised in a vector format according to:

$$\boldsymbol{\varepsilon} = \begin{bmatrix} \varepsilon_{LL} & \varepsilon_{RR} & \varepsilon_{TT} & \gamma_{LR} & \gamma_{LT} & \gamma_{RT} \end{bmatrix}^T \tag{2.3}$$

$$\boldsymbol{\sigma} = \begin{bmatrix} \sigma_{LL} & \sigma_{RR} & \sigma_{TT} & \tau_{LR} & \tau_{LT} & \tau_{RT} \end{bmatrix}^T \tag{2.4}$$

The symmetric elastic flexibility tensor $\mathbf{C}$ describes the connection between the stresses and the strains, and is for an orthotropic material with material orientations LRT rewritten in matrix format as:

$$\mathbf{C} = \begin{bmatrix} \frac{1}{E_L} & -\frac{\nu_{LR}}{E_R} & -\frac{\nu_{LT}}{E_T} & 0 & 0 & 0 \\ -\frac{\nu_{RL}}{E_L} & \frac{1}{E_R} & -\frac{\nu_{RT}}{E_T} & 0 & 0 & 0 \\ -\frac{\nu_{TL}}{E_L} & -\frac{\nu_{TR}}{E_R} & \frac{1}{E_T} & 0 & 0 & 0 \\ 0 & 0 & 0 & \frac{1}{G_{LR}} & 0 & 0 \\ 0 & 0 & 0 & 0 & \frac{1}{G_{LT}} & 0 \\ 0 & 0 & 0 & 0 & 0 & \frac{1}{G_{RT}} \end{bmatrix} \tag{2.5}$$

where E_i, ν_{ij}, and G_{ij} specify the three moduli of elasticity, six Poisson's ratios, and three moduli of rigidity. Thus, the elastic behaviour of wood can be described by twelve constants, of which nine are considered independent since the moduli of elasticity E_i and Poisson's ratios ν_{ij} are dependent according to:

$$\frac{\nu_{ij}}{E_i} = \frac{\nu_{ji}}{E_j}, \qquad i \neq j \qquad i,j = L, R, T \tag{2.6}$$

Table 2.1: Stiffness properties presented for Scots pine, birch and structural timber of different strength classes (C16, C24, C40).

Species	ref	ρ	u	E_L	E_R	E_T	G_{LR}	G_{LT}	G_{RT}	ν_{RL}	ν_{LR}	ν_{LT}	ν_{TL}	ν_{RT}	ν_{TR}
Scots pine	[5]	550	10	16 300	1 110	570	1160	680	66	0.42	0.038	0.015	0.51	0.31	0.68
Birch	[5]	620	9	16 300	1 110	620	1180	910	230	0.49	0.034	0.018	0.43	0.38	0.78
Timber, C16	[8]		12	8 000	270	270	500	500							
Timber C24	[8]		12	11 000	370	370	690	690							
Timber, C40	[8]		12	14 000	470	470	880	880							

In Table 2.1, elastic properties for Scots pine, birch and structural timber of different strength classes (according to EN 338:2016 [8]) are presented. Generally, the modulus of elasticity is much lower perpendicular to the grain than parallel to the grain, and their interrelation can be described by $E_L >> E_R > E_T$. The variation of stiffness in the RT-plane is illustrated for Scots pine, birch and spruce in Figure 2.7 [9].

Strength properties

The typical behaviour of wood subjected to uni-axial loading in the grain direction (LL) or perpendicular to the grain (TT, RR) is schematically illustrated in Figure 2.8, for compression and tension. The point where non-linearities are introduced is referred to as the limit of proportionality. In tension along the grain, this point occurs at about 60% of the ultimate load, whereas it appears much earlier for compression along the grain (30% to 50% of the ultimate load). In compression, the behaviour of the material is ductile, with a clear plateau where, crushing and buckling of the wood takes place. For tension, the failure is often more brittle. The tensile strength parallel to the grain is generally much higher compared to the

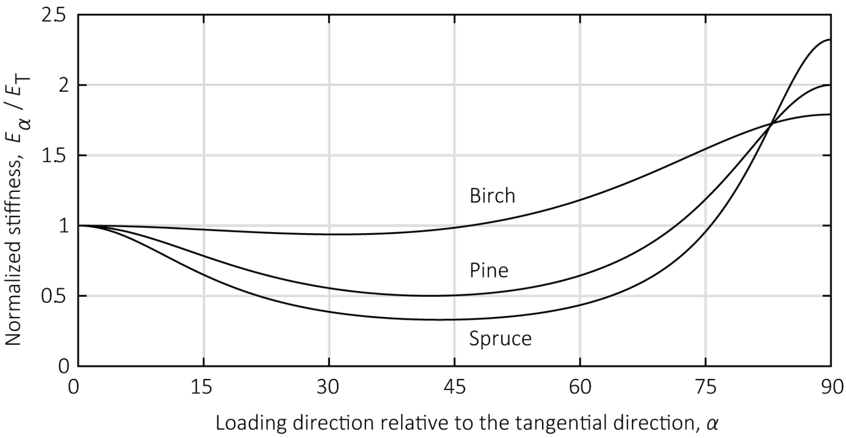


Figure 2.7: Normalized stiffness in the tangential-radial plane, depending on loading direction α relative to the tangential direction. Modified after [9].

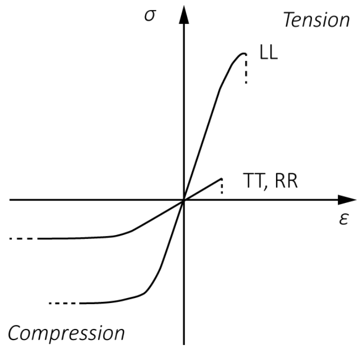


Figure 2.8: Schematic illustration of stress versus strain for uni-axial loading of wood perpendicular (TT, RR) and parallel (LL) to the grain. Modified after [10].

tensile strength perpendicular to the grain as well as the shear strength along the grain.

Density and moisture-dependency

Both stiffness and strength properties are affected by several factors, depending on both the structure of the material and environmental circumstances. Typically, a high correlation is found between density and most mechanical properties, where an increased density corresponds to increased stiffness and strength. For a change in moisture content, no major effects are recognized above the FSP. However, below the FSP, a decreased moisture content generally results in increased stiffness and strength; As water molecules are subtracted, the bond between the microfibrils within the cell wall is strengthened. The largest impact of the moisture content is observed for compression strengths, whereas tensile strengths generally demonstrate a less pronounced moisture-dependency.

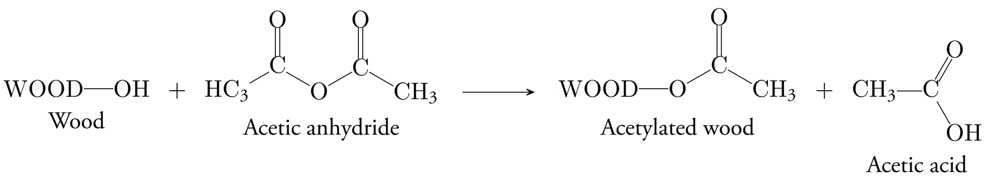
2.2 ACETYLATION

> Combining all of the art and science of wood recorded from ancient times to the present, we have discovered that if you change the chemistry of wood, you change its properties and, therefore, you change its performance. From this foundation, the science of chemical modification of wood was born.
>
> R.M. Rowell

Due to the hygroscopic nature of wood, many wood species used for timber structures demonstrate poor dimensional stability. The long-term presence of moisture further makes timber structures vulnerable to biological decay. Wood modification methods have been developed to improve upon these two undesired characteristics, partly driven by environmental concerns in limiting the use of toxic preservative treatments. By changing the chemical composition of the cell wall polymers, modification methods have proven successful in limiting the hygroscopic characteristics of wood. At the end of its life cycle, chemically modified wood can be disposed without presenting any additional environmental hazards as compared to unmodified wood [11]. Acetylation is one of the most studied chemical modification methods. It was introduced in Germany in 1928 by Fuchs [12], and has today been successfully introduced on the market. The following section focuses on the process of acetylation, how the chemical composition of the cell wall is altered, and how this, in turn, affects physical and mechanical properties of wood. The section is based on [11, 13–15].

2.2.1 Chemical process of acetylation

Acetylation is based on the reaction between acetic anhydride and the cell wall polymeric hydroxyl groups, forming an ester bond between the hydroxyl and the acetyl group, as well as the by-product acetic acid:



The reaction is a single-addition reaction, meaning that one acetyl group is bound to one hydroxyl group. It can take place with the acetic anhydride in liquid or the vapour phase. To express the extent of the reaction of acetic anhydride, the acetyl weight percentage gain (WPG) is determined according to:

$$\mathrm{WPG} = \frac{m_1 - m_0}{m_0} \times 100 \tag{2.7}$$

where m_1 and m_0 represent the oven-dry weight of the wood after and prior to the modification, respectively.

The reactivity of acetic anhydride and wood depends on several parameters: The *permeability* of the wood substrate dictates the accessibility of the reagent; High *moisture contents* improve the accessibility of the reagent, but since water molecules also react with the acetic anhydride, it causes a loss of reagent; High *temperatures* result in faster reactions, but the temperature must be limited since cell wall degradation becomes significant above 120°C; *Catalysts* can be used to function as swelling agents and facilitate the reaction, but are not considered suitable for large-scale industrial processes [13]. Moreover, it is important to attain a consistent and uniform treatment in the process of acetylation. In case of an uneven distribution of acetyl groups, the material will not be protected against biological decay to the same extent. As a consequence, distortion of the material may cause internal stresses, possibly resulting in the formation of cracks. Key parameters for a uniform treatment are time, temperature and pressure [16,17]. These parameters should thus be chosen based on a deep knowledge of the specific wood species.

2.2.2 Properties of acetylated wood

Physical properties

As the chemical composition of the cell wall polymers is altered in acetylated wood, so are its physical properties. Acetylated wood exhibits an increased density, due to acetyl groups occupying the hydroxyl groups. At a WPG of 15%–20%, the increase in dry mass is approximately 5%–20%. The added acetyl groups will also affect the wood volume. At a WPG above 20%, the oven-dry wood volume of an acetylated sample is approximately equal to the green volume of the same unmodified wood sample. Acetylated wood can, thus, be considered to be in a permanently swollen state. Consequently, the cross-section of an unmodified sample will contain about 10% more fibres per cross-section area compared to an equally sized but acetylated sample [15].

To account for the increase in dry mass, the EMC of acetylated wood is sometimes expressed by the reduced equilibrium moisture content EMC_R. The EMC_R represents the weight of the water contained, divided by the oven-dry weight of the wood substance prior to acetylation, m_0:

$$\mathrm{EMC}_R = \frac{m_2 - m_1}{m_0} \tag{2.8}$$

where m_2 is the weight of the acetylated wood in equilibrium with the ambient temperature and relative humidity, and m_1 the oven-dry weight of the acetylated wood.

Due to less accessible hydroxyl groups and more hydrophobic fibres in acetylated wood, it exhibits a reduced EMC compared to unmodified wood. In Figure 2.9, sorption isotherms for unmodified and acetylated wood are shown, clearly demonstrating the reduced water sorption. A parameter describing the reduced EMC is the moisture exclusion efficiency MEE, defined as:

$$\mathrm{MEE} = \frac{\mathrm{EMC}_0 - \mathrm{EMC}_1}{\mathrm{EMC}_0} \tag{2.9}$$

where EMC_0 and EMC_1 are the EMC for unmodified and acetylated wood, respectively. Other characteristics altered by the acetylation process are e.g. a reduced FSP and lower permeability of gases, depending on the WPG of the sample considered.

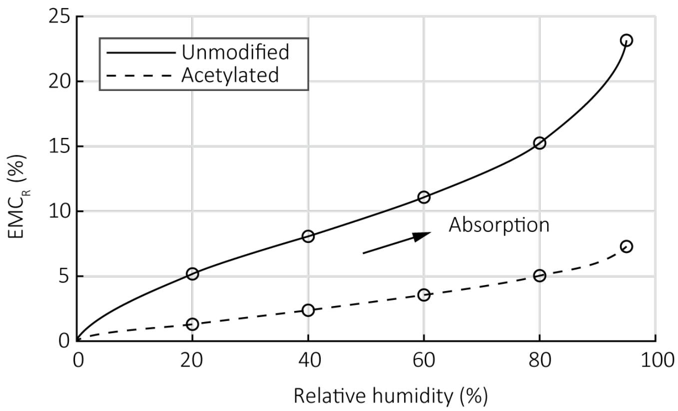


Figure 2.9: Absorption isotherms for unmodified and acetylated Scots pine, indicating the reduced water sorption of acetylated wood.

The dimensional stability of acetylated wood has proven to be superior compared to unmodified wood. This changed behaviour is owed to the fact that acetylated wood will already be in a swollen state, thus, the addition of water molecules will not affect the swelling/shrinking in the same way. Moreover, tests on biological decay have shown an improved resistance to biological degradation. For compilations of studies see e.g. [14, 18].

Mechanical properties

Mechanical properties of wood depend on both moisture content and density. Since these characteristics are changed for wood when acetylated, an impact on the mechanical properties of wood can be expected. Several studies have compared mechanical properties of unmodified and acetylated samples in equilibrium with equal relative humidity levels. However, as outlined by Rowell [15], due to "*the great effect of moisture content on mechanical properties, it is misleading or even invalid, to compare strength properties of control and modified wood since they were tested at different moisture levels*". The comparison of unmodified and acetylated wood of equal dimensions has also been questioned [15], since acetylated wood contains fewer fibres per cross section area. Nevertheless, from the perspective of a structural engineer, the impact in terms of relative humidity and nominal cross section is of utmost importance.

Previous studies of the impact of acetylation on mechanical properties include e.g. an increased wet strength [19], hardness [20, 21] and compressive strength parallel and perpendicular to the grain [20, 21]. Moreover, reduced relative creep under cyclic relative humidity conditions has been found for acetylated wood [22], and a decreased impact on strength values in moist conditions [23]. Conversely, significant reductions of the shear strength have been demonstrated for various wood species [20], and a reduced fracture energy in mode I has been found for acetylated spruce [1]. Other, well-studied, mechanical properties include the modulus of elasticity (MOE) and modulus of rupture (MOR) in bending, where various results have been presented [20–22, 24]. Overall, it is difficult to draw general conclusions based on performed studies, due to differences in testing procedures, acetylation processes, conditioning, and various wood species.

2.3 THE FRACTURE BEHAVIOUR OF WOOD

> You look closely enough, you'll find that everything has a weak spot where it can break, sooner or later.
>
> Anthony Hopkins

In practical timber engineering design situations, assumptions of a linear elastic material behaviour and stress-based failure criteria are often used to assess the capacity of a member or a structure. However, when large stress concentrations occur, this approach is likely to yield poor predictions of the load-bearing capacity. For the case of stress singularities, linear elasticity and stress-based failure criteria are of no use and the load-bearing capacity can then instead be assessed by the use of fracture mechanics. Fracture mechanics deals with separation of material, i.e. the formation and propagation of cracks. Abandoning traditional stress-based failure criteria and resorting to fracture mechanics is often necessary when notches, holes, cracks or sharp stress gradients are present in the structure to be analysed. The following section is based on [10, 25–28].

2.3.1 Crack orientations and modes of loading

As a consequence of the orthotropic nature of wood, fracture properties depend on the orientation of the crack growth with respect to the material directions. Six possible crack propagation systems are illustrated in Figure 2.10a, defined by (1) the normal to the fracture surface, and (2) the direction of the crack length extension. In structural applications, using conventional glulam and timber elements, crack propagation along the grain, i.e. the TL and RL orientations, tend to be most relevant for the load-bearing capacity. Orientations RT and TR are sometimes relevant for joints (e.g. nailed plates) and cross-laminated timber (cracking in transverse layers).

Fracture characteristics are further dependent on how the material is loaded, where three types of relative displacements are distinguished. These are commonly referred to as modes of fracture, and are illustrated in Figure 2.10b. Mode I represents the so called opening mode, consisting of pure tensile stresses oriented in the direction of the normal to the crack surface. Mode II represents the sliding mode, where shear stresses act parallel to the direction of the crack extension. Mode III is often referred to as the tearing mode, and represents out-of-plane shear stresses acting perpendicular to the direction of the crack extension. In general, loading

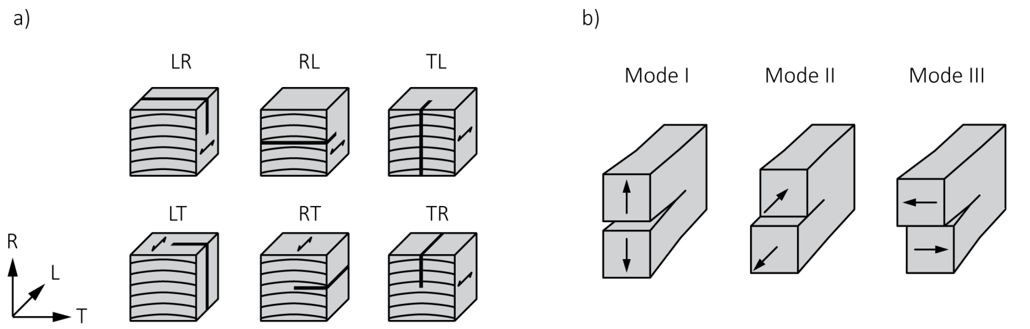


Figure 2.10: Six possible crack propagation systems for wood (a), indexed by the normal to the fracture surface and the direction of the crack length extension, based on the longitudinal (L), radial (R) and tangential (T) directions. Modes of fracture depending of relative displacements I, II, and III (b). Modified after [10].

is a combination of these distinct modes, and such loading is referred to as mixed loading. For timber structures, a combination of mode I and mode II often occurs, e.g. at holes, notches and in dowel-type connections loaded perpendicular to the grain.

2.3.2 Linear elastic fracture mechanics

Linear elastic fracture mechanics (LEFM) was originally developed for isotropic materials, assuming an ideally linear elastic material behaviour. It is a method used to predict the load level at which a pre-existing crack will propagate. The combined requirements of linear elasticity and the existence of a sharp crack, result in singular stress and strain values at the crack tip. Although this lacks physical basis, it is accepted within the theory of LEFM as long as the size of fracture process region (the region ahead of the crack tip where de-bonding occurs) is small compared to the length of the initial crack and distances to supports, loads and boundaries. Since stress and strain values approach infinity, other evaluation quantities must be used. Characterising LEFM quantities are the energy release rate G, the stress intensity factor K, and their corresponding critical values.

The stress intensity approach

The stress intensity factor K is used within LEFM to predict the stress intensity in the vicinity of a sharp crack tip. It is a function of the specimen geometry, applied load and crack length, and a general definition for K is given by:

$$K = \beta\sigma\sqrt{a} \tag{2.10}$$

where β is a dimensionless parameter depending on the geometry, σ the far field stress, and a the crack length. It is separated into the different modes of loading, denoted by K_I, K_{II}, K_{III}. The stress intensity factor K represents the load intensity and can be compared to the corresponding critical value, K_c, to determine at what load crack growth will occur. The critical stress intensity factor is considered a material parameter. By Equation 2.10, it is apparent that by knowing the critical stress intensity factor, one can predict a critical crack length, i.e. the crack length at which crack propagation will occur, for a given stress level.

The energy balance approach

Crack propagation analysis may also be formulated based upon Griffith's energy balance approach, considering the change in total potential energy of a system. For crack propagation to occur, the potential energy Π must be reduced. The strain energy release rate G is defined as the change in energy at an infinitely small increase in crack area dA by:

$$G = -\frac{\partial \Pi}{\partial A} = -\frac{\partial}{\partial A}(U_e + U_P) \qquad (2.11)$$

where U_e is the elastic strain energy stored in the body, and U_P the potential energy of the loads. The energy release rate at which a crack will grow is referred to as the critical energy release rate, G_c. It is considered a material parameter, depending on material orientations and the mode of loading. For a combined loading scenario, $G = G_I + G_{II} + G_{III}$. The general condition for crack growth can be formulated as:

$$G \geq G_c \qquad (2.12)$$

One straight forward way to perform crack propagation analysis of a material or a structure, is by the compliance method. For a 2d-system of width b, loaded by a single load P, and with an existing crack length a, the energy released during propagation of the crack by a length Δa can be expressed as:

$$W = 0.5P^2(C(a + \Delta a) - C(a)) \qquad (2.13)$$

where $C(a)$ is the compliance (i.e. the inverse of the stiffness) as a function of the crack length a (c.f. Figure 2.11). Thus, the energy release rate becomes:

$$G = \frac{W}{b\Delta a} = \frac{1}{2b}P^2\frac{\Delta C}{\Delta a} \qquad (2.14)$$

which, as Δa approaches 0 results in:

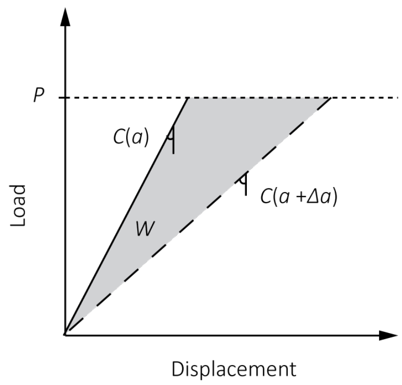


Figure 2.11: Load versus displacement for a linear-elastic structure with crack lengths a and $a + \Delta a$, illustrating the change in compliance C and the energy released during crack propagation W. Modified after [26].

$$G = \frac{1}{2b} P^2 \frac{\partial C}{\partial a} \tag{2.15}$$

Thus, assuming that crack propagation takes place, i.e. $G = G_c$, and by monitoring/calculating $\partial C / \partial a$ it is possible to predict the corresponding critical load P_c if G_c is known, or vice versa.

2.3.3 Non-linear fracture mechanics

LEFM usually provides predictions of sufficient accuracy when the global behaviour of structural sized members, or complete structural systems, are analysed. However, due to the assumption of linear elasticity of the material right until rupture, LEFM may lead to a poor representation of the physical process within the fracture process zone. To account for material non-linearities, including toughening mechanism such as fibre bridging, non-linear fracture mechanics (NLFM) methods can be used. The main advantage of using NLFM is the ability to predict post-peak stress fracture behaviour. Fracture characteristics considered within NLFM are the material strength, the material stiffness and the fracture energy. The fracture energy, G_f, is defined as the energy needed to produce a unit area of traction free crack, i.e. the energy needed to form two separated crack surfaces. The fracture energy G_f and the critical energy release rate G_c share the same units, and if LEFM theory applies and the material has a minimal sized fracture process zone, they are equal, i.e. $G_f = G_c$. However, for quasi-brittle materials, values tend to differ; the fracture energy G_f considers the energy needed for complete separation of two crack surfaces (including strain-softening), while the energy release rate G_c is the energy needed for an incremental crack propagation. G_f is sometimes also referred to as the specific fracture energy.

Cohesive zone modelling

The cohesive zone model, also known as the fictitious crack model, is based on the assumption of a non-linear behaviour within the fracture process zone. The non-linear material behaviour is quantified and described by the softening properties of the material. The softening properties correspond to a stress-deformation based relation, describing how the stress-transferring capability within the fracture process zone is affected as deformations increase. Thus, in cohesive zone modelling, two constitutive relations are applied; one for the material outside the fracture process zone (a regular stress-strain relation), while a stress-deformation relation is applied within the fracture process zone.

To demonstrate the softening properties of a material, a hypothetical experiment can be used (see [28]), considering a homogeneous bar subjected to uniaxial tension applied by displacement control (Figure 2.12). If the test is stable, it is possible to record the complete load-displacement response, including the descending branch until complete fracture. The first part (A in Figure 2.12a) of the load-displacement curve is attributed to the linear elastic response. Thus, the elongation of the bar δ is given by the elastic strain ε and the original length of the bar L_0 ($\delta = L_0\varepsilon$). Assuming that the fracture process zone starts to develop when the peak

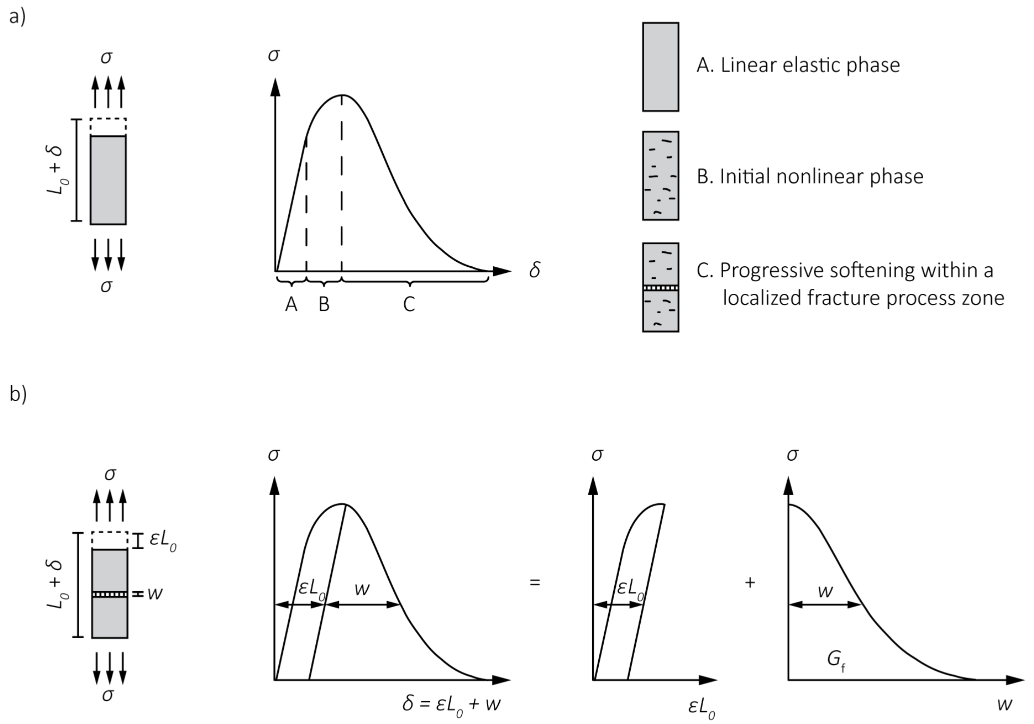


Figure 2.12: Illustration of a stable uniaxial tensile test and corresponding load-displacement response (a). Constitutive equations (σ-ε) and (σ-w) for the linear elastic material and the fracture process zone, respectively (b). Modified after [10].

load is reached, any additional elongation will be due to deformation in the concentrated fracture process zone, and the material outside the process zone will be elastically unloaded (C in Figure 2.12a). Since the stress within the fracture process zone diminishes while deformations increase, it is referred to as softening of the material. After the peak load, the total elongation of the bar is the sum of the elongation due to elastic straining along the bar ($L_0\varepsilon$), plus the additional deformation within the fracture process zone (w). From the load-displacement response, the softening properties (σ-w) can be extracted as shown in (Figure 2.12b). For $w = 0$, the fracture process has not been initiated.

Within cohesive zone modelling, the softening properties are considered to be material properties, assumed for each material point on the crack surface. The strength of the material f_t indicates when softening will begin and the area under the σ-w curve is the fracture energy, i.e. the energy required to create a unit area of traction-free crack. For wood, a bi-linear relationship is often assumed.

2.3.4 Fracture characteristics of wood

To determine fracture characteristic of wood, various test methods can be used, depending on mode of loading and crack orientation. Examples of common set-ups used to determine the mode I fracture energy are shown in Figure 2.13, including Compact Tension specimens (CT), Double Cantilever Beams (DCB) and Single Edge Notched Beams (SENB).

In the present research, SENB specimens subjected to three-point-bending have been used to determine the fracture energy in mode I. One of its benefits is its simplicity, as it only requires the evaluation of the energy put into the system by the load applied. On the other hand, the only information obtained is the fracture energy. More sophisticated methods, providing additional information include e.g. the use of digital image correlation (DIC) techniques.

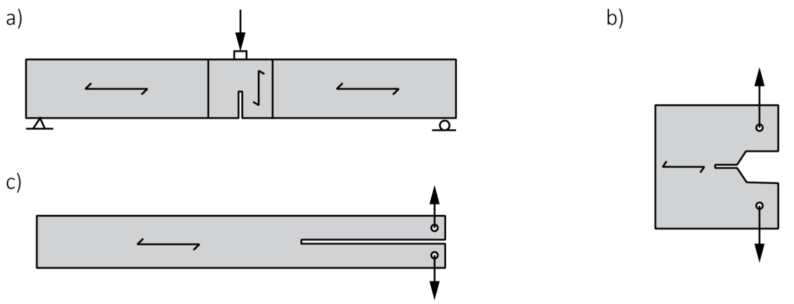


Figure 2.13: Examples of test set-ups to determine the mode I fracture energy: Single Edge Notched Beam (a), Compact Tension specimen (b), and Double Cantilever Beam (c). Modified after [10].

2.4 DOWEL-TYPE CONNECTIONS IN CODES OF PRACTICE

A chain is no stronger than its weakest link

Thomas Reid

To connect members within timber structures, efficient joints are needed. Dowel-type connections are common within timber engineering, since they are easy to produce and assemble. Considering the safety of a structure, ductile failures are favourable, where yielding of metal dowels or embedment failure of the wood are underlying mechanisms. However, brittle failures, resulting in a sudden collapse of a structure, may occur. Brittle failure modes in timber structures may occur due to tensile stresses perpendicular to the grain, shear stresses along the grain, or a combination of both. In general, tension perpendicular to the grain should be avoided in timber design, but this is not always possible – the occurrence of mechanical joints, holes, notches etc., can induce large tensile stresses perpendicular to grain, which may lead to crack initiation and propagation. In the following section, considerations in Eurocode 5 regarding dowel-type connections are briefly introduced. The section is based on [29, 30].

2.4.1 European yield model

The load-bearing capacity of a dowel-type connection is governed by the capacity of the dowel, the embedment strength of the timber, and a possible axial load-bearing capacity of the fastener. For dowels, this axial capacity is known as the rope effect. It enables the transfer of tensile stresses in the dowel, but is often neglected for dowelled connections. In Eurocode 5, the

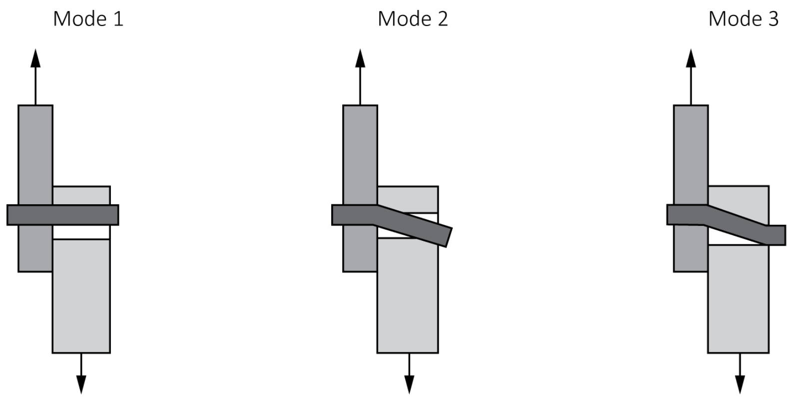


Figure 2.14: Ductile failure modes of dowel-type connections, where mode 1 represents embedment failure of the wood. In mode 2 and mode 3, one or two plastic hinges are formed in the dowel.

load-bearing capacity of dowel-type connections is evaluated based on the European Yield Model (EYM), originating from the Johansen theory. It assumes a ductile failure attributed to yielding of the dowel and/or embedment failure of the wood. Three distinct modes of failure (c.f. Figure 2.14) are identified, based on the formation of plastic hinges in the dowel. For mode 1, no plastic hinges are formed and the capacity is solely estimated based on the embedment strength and the geometry of the joint. For mode 2 and mode 3, one or two plastic hinges are formed, and the load-bearing capacity depends on yielding of the dowels as well as the embedment strength of the wood. All modes of failure must be evaluated, and the lowest capacity should be regarded.

The embedment strength resembles the compression strength around the dowel. The design criterion in Eurocode 5 to determine the embedment strength is based on the dowel diameter and the density of the timber. For loading at an angle to the grain, the embedment strength is reduced based on a correction factor, depending on whether the material of the connection is a softwood or a hardwood.

2.4.2 Brittle failure modes

The EYM assumes ductile failures due to yielding of the fasteners and/or embedment failure of the timber members. However, due to stress concentrations e.g. caused by closely placed fasteners, or fasteners close to the end or the edge of the timber members, premature brittle failure modes may occur. For loading parallel to the grain, possible brittle failure modes include *row shear*, *block shear*, *plug shear* and *splitting*, illustrated in Figure 2.15. For loading at an angle to the grain, there is always a risk of splitting.

To limit the risk of brittle failures, recommended minimum end- and edge-distances are defined in Eurocode 5. The interaction between fasteners, which may cause splitting between fasteners,

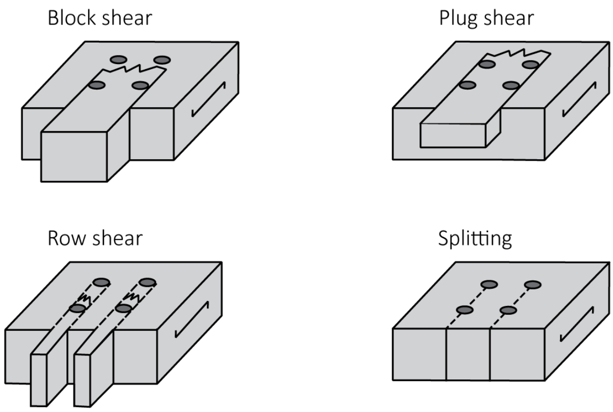
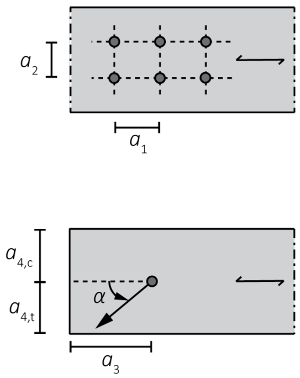


Figure 2.15: Brittle failure modes of dowel-type connections.



Spacings	a_1:	$0 \leq \alpha \leq 360$	$(3 + 2\lvert\cos\alpha\rvert)d$
	a_2:	$0 \leq \alpha \leq 360$	$3d$
Loaded end	$a_{3,t}$:	$-90 \leq \alpha \leq 90$	max [7 d; 80 mm]
Unloaded end	$a_{3,c}$:	$90 \leq \alpha < 150$	$a_{3,t}\lvert\sin\alpha\rvert$
		$150 \leq \alpha < 210$	max [3.5 d; 40 mm]
		$210 \leq \alpha \leq 270$	$a_{3,t}\lvert\sin\alpha\rvert$
Loaded edge	$a_{4,t}$:	$0 \leq \alpha \leq 180$	max[$(2 + 2\sin\alpha)d$; $3d$]
Unloaded edge	$a_{4,c}$:	$0 \leq \alpha \leq 180$	$3d$

Figure 2.16: Spacings between dowels, as well as end- and edge-distances for dowels, where *d* is the diameter of the dowel.

is restricted by recommended spacings. These recommendations are presented in Figure 2.16, and as indicated, they depend on the diameter of the dowel d and the direction of the load relative to the grain α. Worth noting is that these design criteria are based on implicitly assumed material characteristics.

In Annex A of Eurocode 5, methods to quantify the resistance against block and plug shear failure are provided. However, for loading parallel to the grain, no explicit design recommendations are included to verify the risk of splitting, nor of row shear failure. For loading perpendicular to the grain, a criterion to estimate the splitting capacity is included: For a dowel connection according to Figure 2.17, the characteristic shear force capacity is given by:

$$F_{90,Rk} = 14b\sqrt{\frac{h_e}{1 - h_e/h}} \tag{2.16}$$

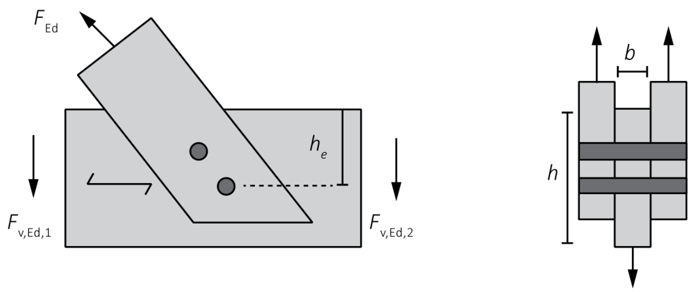


Figure 2.17: Dowel-type connection loaded perpendicular to the grain.

Based on this criterion, the shear force capacity solely depends on the geometry of the connection, and the criterion does not include any material property parameters. The original format of the design equation is derived from linear elastic fracture mechanics, and has been presented as [31]:

$$F_{90,Rk} = b\sqrt{\frac{G_c G}{0.6}}\sqrt{\frac{h_e}{1 - h_e/h}} \tag{2.17}$$

where G_c is the critical energy release rate and G the longitudinal shear modulus. It is apparent that assumptions regarding linear elastic fracture properties are already imposed in the simplified criterion, which is based on results from tests conducted on softwoods.

3 Overview of present work

3.1 GENERAL REMARKS

This chapter aims at giving an overview of the work presented in appended papers A–C. The presentation is divided into two sections: The first section covers material characterisation of unmodified and acetylated clear wood, while the second focuses on structural timber, considering the effect of acetylation on dowel-type connections. Since the work has been focused on experimental characterisation, numerical analyses are not covered in the appended papers. The work has, however, included numerical modelling of test set-ups, which are briefly reviewed as a part of the first section.

3.2 MATERIAL CHARACTERISATION

Before the use of acetylated Scots pine and birch can be realised in structural applications, it is essential to understand also how the material is affected on a clear wood level. Previous studies on the effects of acetylation on various wood species have been conducted with regard to basic mechanical properties, such as bending stiffness and strength, while fewer studies are available on the impact on fracture characteristics. The studies presented in papers A–B, reviewed in this section, focus on fracture characteristics, which here refers to material parameters that influence the brittleness of the material, i.e. strength, stiffness and fracture energy.

3.2.1 Strength, stiffness and fracture energy

In the study presented in paper A, unmodified and acetylated Scots pine (*Pinus sylvestris*) was investigated, conditioned until equilibrium at 20°C and 60% relative humidity. The fracture energy was determined for mode I loading in tension perpendicular to the grain, with a TL-oriented crack propagation system (Figure 3.1a). Moreover, the modulus of elasticity in compression along the grain (Figure 3.1b), and the tensile strength perpendicular to the grain (Figure 3.1c) were determined. The fracture energy was determined for specimens consisting of

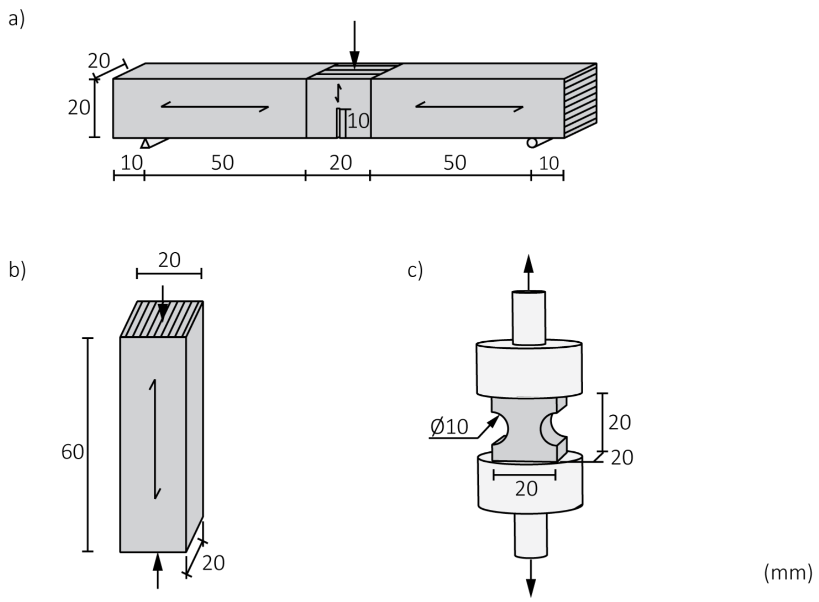


Figure 3.1: Test set-ups used in paper A for determining the mode I fracture energy perpendicular to the grain (a), modulus of elasticity in the fibre direction (b) and tensile strength perpendicular to the grain (c).

either sapwood or heartwood, whereas the modulus of elasticity and the tensile strength were only determined for sapwood.

Compiled results from paper A are presented in Figure 3.2, where differences in mean fracture energy, modulus of elasticity, and tensile strength for acetylated wood relative to unmodified wood are presented in terms of percentage change, along with confidence intervals of the difference. The most important finding of the study was the increased brittleness of acetylated wood compared to unmodified wood; when conditioned at 20°C and 60% relative humidity, the fracture energy of acetylated wood is 36%–50% lower than for unmodified wood. No significant effect of the acetylation process on the modulus of elasticity, nor on the tensile strength was found.

In paper A, possible causes of the lower fracture energy of acetylated wood are discussed. Possible reasons for the increased brittleness that are highlighted include the fewer fibres per cross-section area, and degradation of the cell wall polymers, caused by elevated temperatures during the acetylation procedure. Another possible cause is the reduced hygroscopicity of acetylated wood. Since acetylated wood has a lower moisture content compared to unmodified wood when conditioned to equilibrium at equal temperature and relative humidity, the lower fracture energy could merely be a consequence of a drier, hence more brittle, material. This hypothesis resulted in further research, examining the moisture-dependency of the fracture energy for unmodified and acetylated wood (see section 3.2.3).

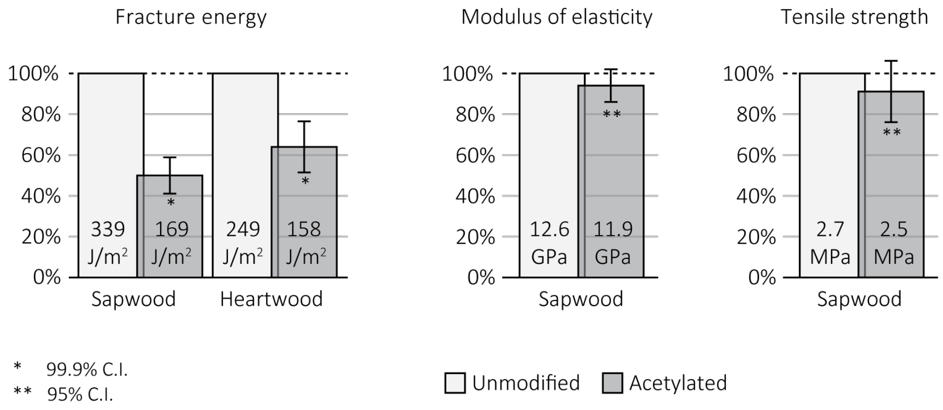


Figure 3.2: Mean values of fracture energy, modulus of elasticity, and tensile strength for acetylated wood relative to unmodified wood, along with confidence intervals (C.I.) of the difference.

3.2.2 Material model calibration

The test set-up used to experimentally determine the fracture energy was examined by finite element analysis. The aim was to calibrate proper material models, and to study the influence of different strain-softening models in terms of type of σ-w curve (c.f. Figure 3.3). To model the non-linear behaviour within the fracture process zone, a cohesive zone approach was used. Numerically, this was done by introducing non-linear spring elements along a predefined crack path, aligned with the initial notch. Cohesive interaction properties were assigned to the non-linear spring elements, representing the strain-softening properties of the material.

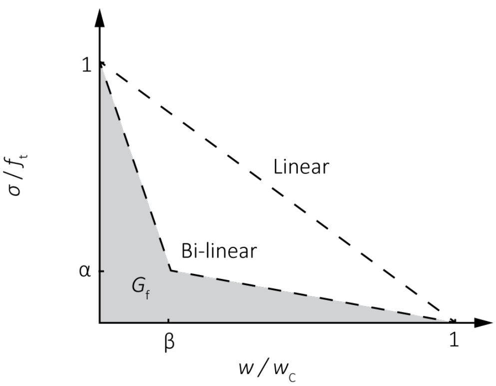


Figure 3.3: Normalised softening models for a linear or a bi-linear softening, where f_t is the tensile strength and w_c the deformation at which the stress equals zero, $\sigma = 0$.

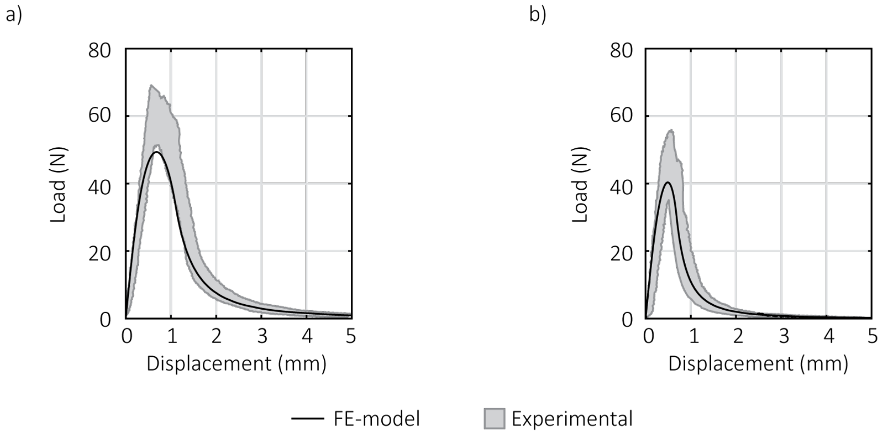


Figure 3.4: Load-displacement responses from paper A and from the numerical analysis (FE-model), presented for unmodified (a) and acetylated Scots pine (b).

One drawback with the the method used in papers A–B is the limited information obtained, i.e. only the fracture energy is evaluated, and no additional information about the softening behaviour of the material is acquired. Assuming a linear strain-softening and implementing the experimentally found mean values of f_t, G_f, and E_L (see Figure 3.2), results from the numerical analysis as well as load-displacement responses from paper A are shown in Figure 3.4. To further examine how the type of softening-curve (e.g. linear or bi-linear) impacts the load-displacement response, a parameter study was conducted. The apparent bending strength, f_f, is here defined by:

$$f_f = \frac{M_{max}}{W} \tag{3.1}$$

where W is the elastic section modulus, and $M_{max} = P_{max}L/4$. P_{max} is evaluated by the peak load of the load-displacement response, and L is the span between the supports (c.f. Figure 3.1a). For a linear strain-softening and a constant tensile strength (3 MPa), an increased fracture energy (Figure 3.5a) corresponded to an increased apparent bending strength of the specimen (Figure 3.5c). Increasing the fracture energy by the same magnitude, but by introducing a bi-linear softening model (Figure 3.5b), the peak load remained constant (Figure 3.5d). It can be concluded that the slope of the first part of the strain-softening curve, attributed to micro-cracking, is decisive for the apparent bending strength.

A structure can be classified by its brittleness ratio according to d/L_{ch} where d considers the size of the structure, and L_{ch} is a characteristic length defined by the material properties according to:

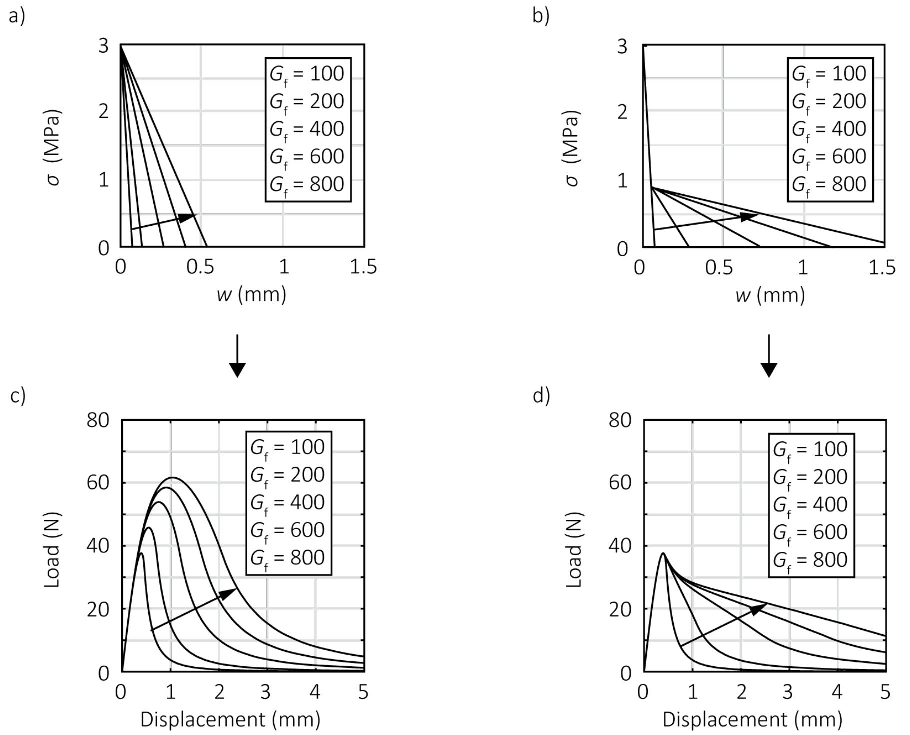


Figure 3.5: Softening properties and load-displacement responses for increased fracture energy and a linear strain softening in (a) and (c), or by introducing a bi-linear strain softening in (b) and (d).

$$L_{ch} = \frac{E_L G_f}{f_t^2} \tag{3.2}$$

When analysing a structure with a small brittleness ratio, the load-bearing capacity will approach the ideally-plastic solution (corresponding e.g. to infinitely large fracture energy G_f). Oppositely, having a large brittleness ratio, methods based on LEFM will be applicable. A study of three different softening models was performed, to analyse the significance of the impact of the type of softening behaviour in the region between an ideally-plastic and a LEFM solution. One linear model, and two bi-linear models ($\alpha = \beta = 0.2$ and $\alpha = \beta = 0.8$, c.f. Figure 3.3) were investigated, in which the tensile strength and fracture energy were kept constant (Figure 3.6a), while varying the dimension *d*. The normalized apparent bending strength versus brittleness ratio was evaluated. Results presented in Figure 3.6b show that the models differ within a certain range of the brittleness ratios, where model A would yield lower strength values than the linear strain-softening, and model B the opposite. For a low brittleness ratio all solutions approach the plastic solution, while for a large brittleness ratio, the LEFM solution is approached.

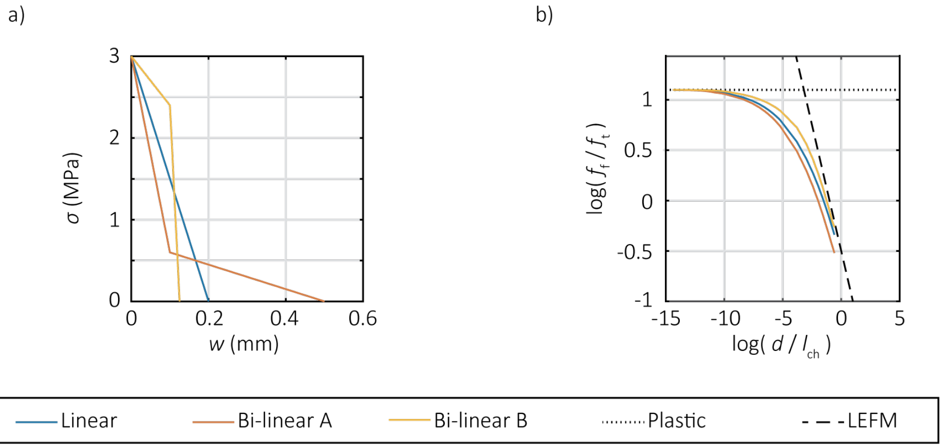


Figure 3.6: Analysed softening models (a) and corresponding normalized apparent bending strength versus brittleness ratio, along with the plastic response and the linear elastic fracture mechanics (LEFM) response (b).

3.2.3 Moisture-dependency

The aim of the study presented in paper B was to investigate if the increased brittleness for acetylated wood (found in paper A) was merely a consequence of the lower moisture content. The moisture-dependency of the fracture energy was studied for unmodified and acetylated Scots pine (*Pinus sylvestris* L.) and birch (*Betula pendula* Roth). Specimens were conditioned at 20°C and relative humidity levels of 20%, 75%, and 97%, using saturated salt-solutions (Figure 3.7a). Additionally, oven-dry (105°C) and fully water-saturated samples (Figure 3.7b) were included in the study. The fracture energy in mode I loading perpendicular to the grain was determ-

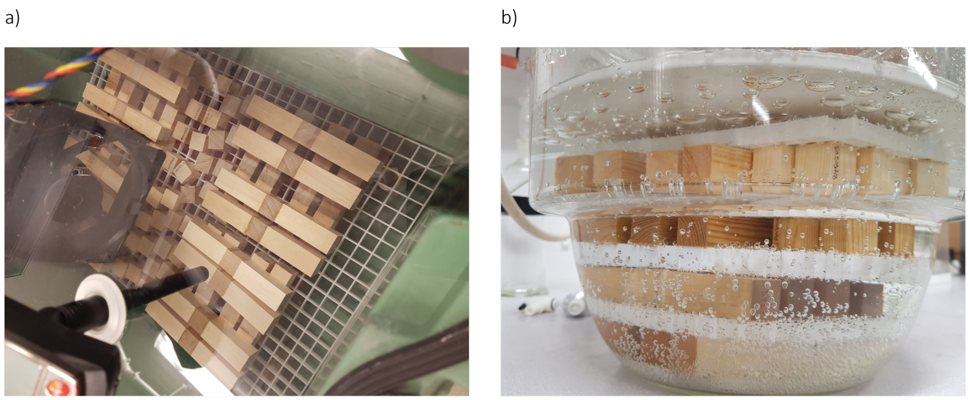


Figure 3.7: Conditioning of specimens using saturated salt-solutions (a) and vacuum-saturation for a well-defined, fully-saturated state (b).

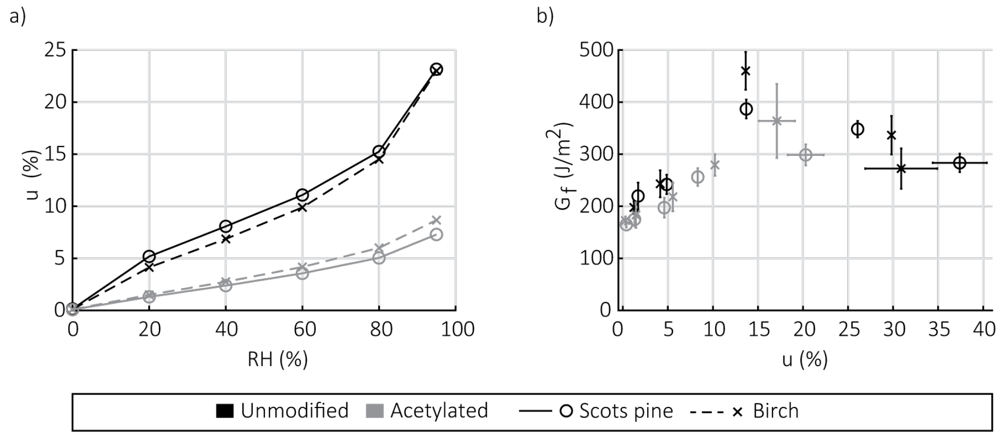


Figure 3.8: Absorption isotherms (a) and fracture energy versus moisture content (b) for unmodified and acetylated Scots pine and birch. Markers denote mean values and error bars standard deviation.

ined, using the standard method presented in paper A (c.f. Figure 3.1a). Moreover, absorption isotherms at 20°C were determined based on a sample equilibrated at relative humidity levels of 0%, 20%, 40%, 60%, 80%, and 95%.

In Figure 3.8a, absorption isotherms are presented for unmodified and acetylated Scots pine and birch, clearly demonstrating the reduced water-sorption of acetylated wood. The relation between moisture content and fracture energy is presented in Figure 3.8b. For moisture contents below 15%, the fracture energy increased with increasing moisture content for both unmodified and acetylated wood. For unmodified wood, a peak in fracture energy was identified around 12%–18% moisture content, possibly attributed to hemicellulose reaching the glass transition point. Further, results corresponding to moisture contents below 15% were investigated by analysis of covariance, indicating a significant difference in moisture-dependency between unmodified and acetylated wood. The test results suggested that the fracture energy was lower for acetylated wood compared to unmodified wood at similar moisture content.

In design codes, modification of material parameters to account for different climate conditions are often based on ambient relative humidity and temperature. Thus, it is important to understand how characteristics of the material depend on relative humidity. The percentage difference in fracture energy for acetylated wood relative to unmodified wood is presented in Figure 3.9, along with 95% confidence intervals of the difference. As shown, acetylated wood demonstrated a significantly lower fracture energy at humidity levels up to 97%. No significant difference was found for water-saturated samples. It was concluded that the lower fracture energy of acetylated wood compared to unmodified wood at equal relative humidity levels can to a certain extent be explained by a combination of reduced hygroscopicity and the moisture-dependency of the fracture energy.

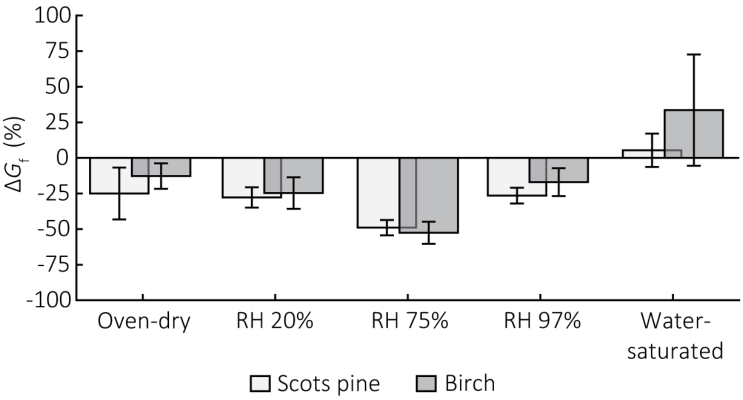


Figure 3.9: Difference in mean fracture energy for acetylated wood relative to unmodified wood for Scots pine and birch. Error bars denote 95% confidence intervals.

3.3 STRUCTURAL APPLICATIONS OF ACETYLATED WOOD

For acetylated Scots pine and birch to be safely used in structural applications, existing design regulations must be verified. The aim of the study presented in paper C was to investigate if the current design provisions of Eurocode 5 can be safely applied to acetylated Scots pine, or should be revised to account for the increased brittleness of such wood.

3.3.1 Dowel-type connections made from acetylated wood

The study presented in paper C examined the effects of acetylation on single-dowel connections. The investigated material consisted of unmodified and acetylated Scots pine, conditioned until equilibrium at 20°C and 60% relative humidity. Dowel-type connections were tested in loading parallel and perpendicular to the grain for varying end- and edge-distances. Moreover, the embedment strength parallel and perpendicular to the grain was determined according to test standard [32].

Compiled results from paper C are presented in Figure 3.11, along with Eurocode 5 approximations that are based on the density of the material. Worth noting is the increased embedment strength for acetylated wood parallel to the grain. As expected, this resulted in an increased load-bearing capacity parallel to the grain for dowel-type connections made from acetylated wood. However, while Eurocode 5 provided conservative estimations, acetylated wood failed in a brittle manner regardless of the studied end-distance. This indicates that the minimum requirements on end-distances given i Eurocode 5 may need adjustments for acetylated wood loaded parallel to the grain in order to achieve ductile failure modes. An alternative, to account for the increased embedment strength parallel to the grain, would be to use some type of reinforcement to prevent brittle failure modes.

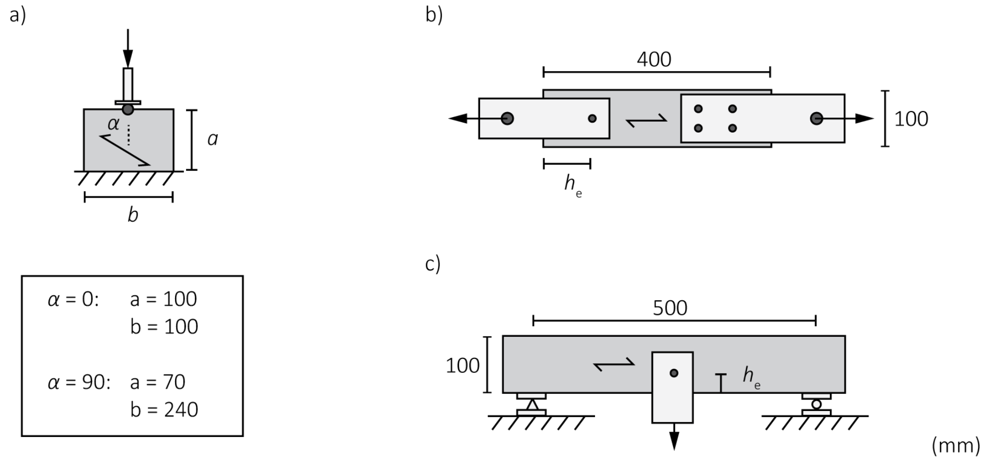


Figure 3.10: Test set-ups used in paper C for determining the embedment strength (a), load-bearing capacity parallel to the grain (b) and perpendicular to the grain (c).

At loading perpendicular to the grain, both unmodified and acetylated wood failed due to splitting, and Eurocode 5 overestimated the capacity. This indicates an insufficiency of current design provisions for loading at an angle to the grain. In agreement with previous studies, indicating a lower fracture energy for acetylated wood, a decreased splitting capacity was observed when compared to unmodified wood.

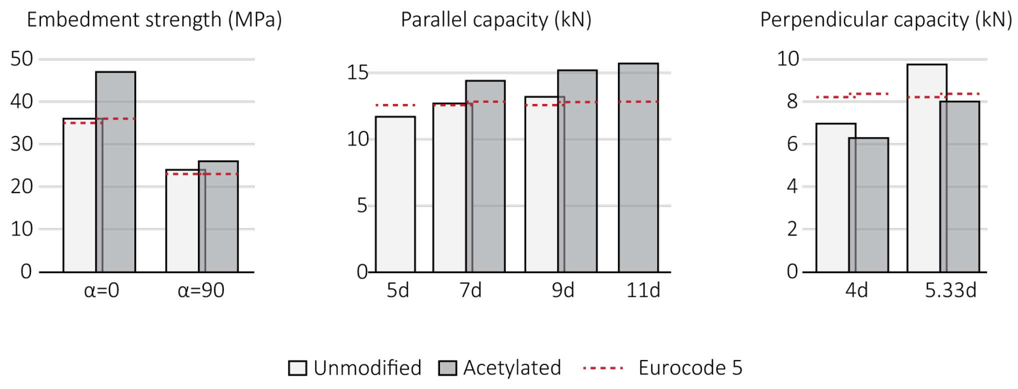


Figure 3.11: Embedment strength (α = direction relative to the grain) and load-bearing capacity at various end-/edge distances (d = diameter of the dowel) for unmodified and acetylated Scots pine, along with Eurocode 5 estimations based on density.

4 Concluding remarks and further work

The general aim of the research has been to investigate the possibility of using acetylated young Scots pine and birch for load-bearing applications. In the following chapter, the most important conclusions and findings from the research presented herein are highlighted. Yet, many related research questions remain unanswered, and thus, proposals for future work are also presented.

4.1 CONCLUSIONS

Material properties

The aim of the research was to investigate the impact of the acetylation process on the fracture characteristics, at a clear wood level. Moreover, the moisture-dependency of the fracture energy was investigated to gain knowledge about the impact for moisture conditions relevant in the design of outdoor load-bearing structures. Research contributions on the effects of the acetylation process on these parameters can be concluded by:

- For Scots pine conditioned until equilibrium at a relative humidity of 60% and a temperature 20°C, the tensile strength perpendicular to the grain and the stiffness parallel to the grain remained unaltered.
- For both Scots pine and birch conditioned at relative humidity levels of 0%–97% and a temperature of 20°C, the fracture energy was significantly lower for acetylated wood (up to –50%).
- The increased brittleness of acetylated wood when compared to unmodified wood at equal relative humidity levels can partly, but not solely, be explained by the markedly reduced hygroscopicity of acetylated wood in combination with the clear moisture-dependency of the fracture energy.

Based on presented findings, an increased brittleness should be regarded for acetylated Scots pine and birch when used in load-bearing structures. In outdoor applications, a fluctuating relative humidity must be accounted for, and thus, the worst impact on the fracture energy (–50%) is suggested to be considered.

Structural applications

In order to add a structural value to acetylated wood, one research aim was to investigate if the current design provisions of Eurocode 5 are valid also for acetylated wood. The load-bearing capacity of dowel-type connections made from unmodified and acetylated Scots pine were examined, and the conclusions from the study can be summarized by:

- An increased embedment strength as well as an increased load-bearing capacity was found for acetylated wood loaded parallel to the grain. Eurocode 5 provided conservative approximations, yet, distinct brittle failure modes were observed for all specimens of acetylated wood, regardless the end-distance.
- The splitting capacity for loading perpendicular to the grain was significantly reduced for acetylated wood, and the load-bearing capacity was in general overestimated by Eurocode 5.

It can be concluded that special attention is required in the design of dowel-type connections made from acetylated wood. To promote ductile failures, current design regulations need to be modified for acetylated wood, e.g. regarding recommended end- and edge-distances, and spacings between fasteners. The increased embedment strength parallel to the grain for acetylated wood yields an increased capacity. However, to make use of this increased capacity, use of reinforcement is recommended to avoid premature brittle failure modes.

4.2 FURTHER WORK

Before realising large-scale use of acetylated Scots pine and birch in load-bearing applications, extensive research is needed. Questions that thus-far remain open include e.g. the compatibility between fasteners and the modified wood material and gluability. This study has indicated the need of consideration of the increased brittleness in the design of dowel-type connections made from acetylated wood. On the other hand, the increased dimensional stability of acetylated wood may limit the risk of cracking (in e.g. joints) and thus limit the number of situations where fracture characteristics would be decisive.

The continued work within this PhD-project will be focused on further examination of structural applications. The long-term effects and impact of moisture on dowel-type connections made from acetylated wood will be objects of further studies. The continued research will also comprise numerical studies to calibrate proper material models to be used for analysis of mechanical joints related to design codes of practice.

References

[1] Reiterer, A., Sinn, G. (2002), *Fracture behaviour of modified spruce wood: a study using linear and non linear fracture mechanics*, Holzforschung **56**(2), 191–198.

[2] Anderberg, S. (2016), *Mechanical properties of chemical modified wood of load-bearing constructions*, Master's thesis, Division of Structural Engineering, Faculty of Engineering LTH, Lund University, Lund.

[3] Chen, C., Kuang, Y., Zhu, S., Burgert, I., Keplinger, T., Gong, A., Li, T., Berglund, L., Eichhorn, S.J., Hu, L., *Structure–property–function relationships of natural and engineered wood*, Nature Reviews Materials **5**, 642–666.

[4] Wiedenhoeft, A.C. (2013), *Structure and Function of Wood*, in: R.M. Rowell (ed.), *Handbook of Wood Chemistry and Wood Composites, Second Edition*, chap. 2, 9–32, CRC Press.

[5] Dinwoodie, J.M. (2000), *Timber; Its Nature and Behaviour, Second Edition*, Taylor & Francis.

[6] Bodig, J., Jayne, B.A. (1982), *Mechanics of wood and wood composites*, Van Nostrand Reinhold, New York.

[7] Côte, W., Kollman, F. (1968), *Principles of Wood Science and Technology*, Springer Berlin, Heidelberg.

[8] EN 338:2016 (2016), *Structural timber – Strength classes*, European Committee for Standardization, Brussels.

[9] Gustafsson, P.J., Danielsson, H. (2013), *Perpendicular to grain stiffness of timber cross sections as affected by growth ring pattern, size and shape*, European Journal of Wood and Wood Products **71**(1), 111–119.

[10] Danielsson, H. (2013), *Perpendicular to grain fracture analysis of wooden structural elements – Models and applications*, Ph.D. thesis, Division of Structural Mechanics, Faculty of Engineering LTH, Lund University, Lund.

[11] Sandberg, D., Kutnar, A., Mantanis, G. (2017), *Wood modification technologies – A review*, iForest - Biogeosciences and Forestry **10**(6), 895–908.

[12] Fuchs, W. (1928), *Genuine lignin. I. Acetylation of pine wood*, Berichte der Deutschen Chemischen Gesellschaft **61**, 948–951.

[13] Hill, C.A. (2007), *Wood Modification: Chemical, Thermal and Other Processes*, vol. 5, John Wiley & Sons.

[14] Rowell, R.M. (2006), *Chemical modification of wood: A short review*, Wood Material Science and Engineering **1**(1), 29–33.

[15] Rowell, R.M. (1996), *Physical and mechanical properties of chemically modified wood*, in: D.N.S. Hon (ed.), *Chemical Modification of Lignocellulosic Materials*, chap. 12, 295–310, Routledge, New York.

[16] Bongers, F., van Zetten, J. (2017), *Consistency of performance of acetylated wood*, in: *The international research group on wood protection, IRG/WP 17-20608*.

[17] Bongers, F., Uphill, S.J. (2019), *The potential of wood acetylation*, in: *7th-International Scientific Conference on Hardwood Processing*, 49–59.

[18] Larsson Brelid, P. (2013), *Benchmarking and state of the art for modified wood*, SP Technical Research Institute of Sweden .

[19] Goldstein, I.S., Jeroski, E., Lund, A., Nielson, J., Weaver, J. (1961), *Acetylation of wood in lumber thickness* .

[20] Dreher, W. (1964), *Mechanical properties of acetylated wood.*, For Prod J. **14**, 66–68.

[21] Bongers, H., Beckers, E. (2003), *Mechanical properties of acetylated solid wood treated on pilot plant scale*, in: *Proceedings of the First European Conference on Wood Modification*, 341–350, Ghent, Belgium.

[22] Epmeier, H., Kliger, R. (2005), *Experimental study of material properties of modified Scots pine*, Holz als Roh-und Werkstoff **63**(6), 430–436.

[23] Marcroft, J., Bongers, F., Perez, F.P., Alexander, J., Harrison, I. (2014), *Structural performance of Accoya® wood under service class 3 conditions*, in: S. Aicher, H.W. Reinhardt, H. Garrecht (eds.), *Materials and Joints in Timber Structures. RILEM Bookseries.*, vol. 9, 615–627, Springer, Dordrecht.

[24] Larsson, P., Simonson, R. (1994), *A study of strength, hardness and deformation of acetylated Scandinavian softwoods*, Holz als Roh-und Werkstoff **52**(2), 83–86.

[25] Smith, I., Landis, E., Gong, M. (2003), *Fracture and fatigue in wood*, John Wiley & Sons.

[26] Serrano, E. (2016), *Rational modelling and design in timber engineering applications using fracture mechanics*, in: *World Conference on Timber Engineering*.

[27] Gustafsson, P.J., *Fracture perpendicular to grain – Structural applications*, 103–130.

[28] Hillerborg, A. (1991), *Application of the fictitious crack model to different types of materials*, in: *Current Trends in Concrete Fracture Research*, 95–102, Springer, Dordrecht.

[29] EN 1995-1-2 (2004), *Eurocode 5, Design of timber structures: Part 1-1, General—-Common rules and rules for buildings* .

[30] Swedish Wood (2015), *Design of timber structures – Structural aspects of timber construction, Volume 1* .

[31] Van der Put, T., Leijten, A. (2000), *Evaluation of perpendicular to grain failure of beams caused by concentrated loads of joints*, in: *Proceedings of CIB-W18/paper*.

[32] ISO 10984-2 (2009), *Timber Structures – Dowel-type Fasteners —- Part 2: Determination of Embedding Strength and Foundation Values*, International Organization for Standardization .

Part II

Appended publications

Paper A

European Journal of Wood and Wood Products
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ORIGINAL ARTICLE

Fracture characteristics of acetylated young Scots pine

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Abstract

A study on the fracture characteristics of unmodified and chemically modified Scots pine (*Pinus sylvestris*) is presented. The investigated material consisted of small-dimension sawn timber originating from young logs (thinnings), aged 30–40 years. The modified samples were acetylated with acetic anhydride in an industrial scale process without the use of any catalyst, reaching an acetyl content of 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 fracture energy for mode I loading in tension perpendicular to the grain was determined using single edge notched beam (SENB) specimens, subjected to three-point bending. Additionally, the modulus of elasticity along the grain and the tensile strength perpendicular to the grain were determined for sapwood specimens. The findings demonstrated a significant decrease (between 36 and 50%) in the fracture energy for the acetylated specimens, compared to the unmodified specimens. No significant effect of the acetylation process on the modulus of elasticity, nor on the tensile strength could be concluded. This study indicates that the acetylation process used results in an increased brittleness for Scots pine. Further studies are needed to analyse why the fracture energy is impaired, and to examine whether and how current timber engineering design provisions can or should be revised to account for the increased brittleness of acetylated Scots pine.

1 Introduction

Softwoods demonstrate low durability and poor dimensional stability when exposed to changes in moisture content, resulting in, for example, crack initiation caused by differential swelling, or loss of strength due to biological degradation. To overcome these drawbacks, without the use of toxic preservatives, different modification methods have been developed. The foundation of chemical modification methods lies in the possibility to change the properties of wood by changing its chemistry and these methods have proven to be successful in limiting the hygroscopic characteristics of wood (Rowell 2006). As there is a change in chemistry of the cell wall polymers, there also is an impact on the physical properties of the wood (Rowell 1996).

Acetylation is one of the most studied chemical modification methods (Rowell 2006) and was introduced in Germany in 1928 by Fuchs (1928). The chemical process of acetylation involves a reaction of acetic anhydride with wood polymers, resulting in the esterification of accessible hydroxyl groups in the cell wall, as well as formation of the by-product acetic acid. The acetylation process is a single-addition reaction, meaning that one acetyl group is bound to one hydroxyl group, without any polymerisation (Rowell 1983). The number of free hydroxyl groups that normally bind water is reduced and substituted by hydrophobic acetyl groups. The combination of a lesser number of accessible hydroxyl groups and more hydrophobic fibres decreases the water sorption. This change in hygroscopicity results in a reduced equilibrium moisture content (EMC) and fibre saturation point (FSP) (Rowell 2006). Furthermore, acetylation impacts the wood volume. For acetylated wood with a weight percentage gain of approximately 20%, the oven-dry wood volume equals the original green volume. Hence, as the wood is in a permanently swollen state, acetylated wood

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exhibits fewer fibres per cross-section area, compared to its unmodified state (Rowell 1996).

Comprehensive studies have shown that acetylated wood exhibits enhanced dimensional stability and improved resistance to biological degradation; for compilations of studies see for example Rowell (1983), Rowell (2006) and Brelid (2013). Changing the chemical constitution of the cell wall polymers may also impact mechanical properties. For modified wood, well-studied mechanical properties are the modulus of elasticity (MOE) and modulus of rupture (MOR), determined through bending tests (see e.g. Dreher et al. 1964; Larsson and Simonson 1994; Bongers and Beckers 2003; Epmeier and Kliger 2005). Findings depend on wood species, climate conditions and utilised acetylation techniques. Previous studies have also reported that acetylated wood demonstrates improved hardness (Dreher et al. 1964; Bongers and Beckers 2003), improved compressive strength parallel and perpendicular to the grain (Dreher et al. 1964; Bongers and Beckers 2003), improved wet compressive strength (Goldstein et al. 1961) and a reduced relative creep, measured under cyclic relative humidity conditions (Epmeier and Kliger 2005). Insignificant effects from acetylation have been concluded regarding the impact strength (Goldstein et al. 1961; Bongers and Beckers 2003), while significant reductions in the shear strength have been demonstrated for various wood species (Dreher et al. 1964).

Whilst the effects of acetylation on dimensional stability, durability and basic mechanical properties have been investigated extensively over the last decades, less research concerning fracture properties has been performed. The occurrence of knots, holes, notches, moisture gradients etc., can induce large tensile stresses perpendicular to grain in structural elements which may lead to crack initiation and propagation (Gustafsson 2003). It is thus important to consider fracture properties when wood is used for structural applications. In the design of mechanical joints, fracture properties related to mode I and II are decisive, for example, when a load is applied at an angle to the grain, or in order to avoid brittle failure modes, such as splitting (Ehlbeck and Görlacher 2017). In particular, dowel-type joints have been researched in terms of brittle failure modes, for which both mode I and mode II fracture energy are of importance for the load bearing capacity (Sjödin and Serrano 2008; Jensen and Quenneville 2011; Cabrero et al. 2019). A study conducted by Reiterer and Sinn (2002) indicates a reduction in the mode I fracture energy with approximately 20% for acetylated spruce. The study also includes fracture characteristics of heat-treated spruce specimens, indicating even larger reductions in the fracture energy, in the order of 50%–80%.

The aim of this study is to further investigate the effects of acetylation on the fracture characteristics of wood, namely Scots pine. In the current paper, the term "fracture characteristics" refers to those material parameters that influence the brittleness of the material, i.e. strength, stiffness and fracture energy. The work includes studies on the fracture energy for mode I loading in tension perpendicular to the grain, modulus of elasticity along the grain and tensile strength perpendicular to the grain for modified and unmodified specimens. The fracture energy is determined for specimens consisting of either sapwood or heartwood, whereas the stiffness and strength are only determined for specimens consisting of sapwood. The investigated material consists of wood from young logs from thinnings. Such wood is rarely used for structural purposes due to its poor durability and poor dimensional stability. By acetylation, the aim is to enhance both durability and dimensional stability. If it is possible to do so, without impairing the mechanical properties, it will enable the use of young Scots pine in outdoor load-bearing applications.

2 Materials and methods

2.1 Materials

This study investigates fracture characteristics of unmodified and modified samples of young Scots pine (*Pinus sylvestris*). The wood was provided by the sawmill Isojoen Saha, located in Finland. The logs used by the sawmill origins within 60 km from the mill, in an area well known for its fast-growing pine. For this study, the term young logs refers to wood from small-dimension sawn timber from thinnings, with an age of approximately 30–40 years, without visible unsound knots. The modification was conducted in a proprietary industrial scale process at Accsys Group's acetylation plant in Arnhem, the Netherlands. The size of the boards was approximately 100 mm × 1000 mm × 30 mm, in the width, length and thickness directions, respectively. The acetylation process involves a reaction of the wood with acetic anhydride at an elevated temperature (approximately 120–130 °C) without the use of any catalyst, and after the reaction, the by-product acetic acid was removed (Rowell and Dickerson 2014). The wood material was acetylated according to the commercial production process for Accoya® radiata pine at Accsys Technologies, according to the standard process (European Patent No. 2818287A1, Giotra 2014). This process is developed for radiata pine, and it should be noted that it was not optimised for Scots pine. All boards used in this study were analysed for acetyl content by near infrared spectroscopy (NIRS) according to the method described by Schwanninger et al (2011). The analysis showed that all boards reached an acetyl content of approximately 20%.

The sawing pattern to extract specimens from each board is illustrated in Fig. 1. From each board three sticks were extracted. The two outermost, furthest from the pith,

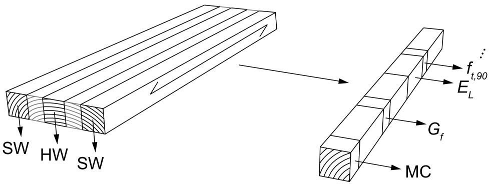

Fig. 1 Sawing pattern for each board, where 3 sticks consisting of either mainly sapwood (SW) or heartwood (HW) were extracted, from which specimens were extracted in the lengthwise direction

contained pure sapwood (SW), based on an ocular distinction between sapwood and heartwood. The inner one contained growth rings closer to the pith, i.e. contained higher fractions of juvenile wood and heartwood. Due to the variability in performance of heartwood as well as juvenile wood, results from these specimens are presented separately from pure sapwood specimens, henceforth referred to as heartwood specimens (HW). Aiming at identifying mechanical properties for clear wood specimens, knots and other imperfections were excluded when extracting specimens from each stick. Specimens originating from the same stick are referred to as nominally equal, due to similarities regarding growth ring orientations and expected limited variation in properties in general.

The fracture energy was determined for both sapwood and heartwood, based on four test groups: unmodified sapwood (USW); modified sapwood (MSW); unmodified heartwood (UHW); modified heartwood (MHW). The modulus of elasticity and the tensile strength were only determined for sapwood, based on two test groups: USW; MSW. The density was determined for each specimen, and the moisture content (MC) for one specimen in each series of nominally equal samples, i.e. one specimen per stick, see Fig. 1. The moisture content was determined by the oven dry method. Prior to testing, all samples were conditioned until equilibrium at a relative humidity (RH) of 60% and a temperature of 20 °C.

2.2 Fracture energy

The fracture energy, G_f, is defined as the energy needed to produce a unit area of traction-free crack and is measured in [J/m^2]. It is the energy dissipated in the fracture process zone, from fracture initiation to creation of traction-free crack surfaces, i.e. when stresses can no longer be transferred between the two fracture surfaces (Gustafsson 2003). There are a number of methods to experimentally determine fracture properties of wood. For mode I (the opening mode), some commonly used methods include the use of compact tension specimens (CT), double cantilever beams (DCB) or single edge notched beams (SENB). In this study, SENB specimens subjected to three-point-bending were used to determine the fracture energy in mode I in tension perpendicular to the grain, according to the standard NT BUILD 422 (1993). The main reason for choosing this test method and the related evaluation methods was its simplicity. Using the approach stated in the standard, no assumptions about, for example, loading/unloading behaviour, bending/shear deformation of the specimen, the shape of the softening curve of the material, the tensile strength perpendicular to the grain, the size of the fracture process zone nor its development are necessary. Only the energy put into the system by the loading applied during the course of the test is evaluated. The most noticeable possible error involved herein is the influence of plastic dissipation at the loading point and at the supports, and plastic dissipation in the compression zone of the specimen. The influence of these sources of error is, however, expected to be negligible in relation to, for example, the variability of G_f between nominally equal specimens, and especially for small specimens like the ones used in the present study (Gustafsson 2003). The drawback when using the Nordtest method is that the information obtained is limited, i.e. only the fracture energy is evaluated. If additional parameters are of interest, for example, the shape of the softening curve of the material, including the strength of the material, more sophisticated (and complex) test and evaluation methods can be used. Such methods include, for example, the use of the so-called R-curve concept, and require additional assumptions regarding the fracture behaviour of the material (see e.g. de Moura et al. 2010; Dourado et al. 2011, 2015; Morel et al. 2005).

The fracture energy was determined for 16 unmodified and 16 modified sapwood specimens, as well as 12 unmodified and 11 modified heartwood specimens. Each specimen consisted of three wood pieces, glued together with polyvinyl acetate (PVAc), with geometry and material orientations defined according to Fig. 2a. The annual rings of the specimens were oriented aiming at a TL crack system, i.e. with the crack propagating in the longitudinal direction (fibre direction), L, and the normal to the fracture surface in the tangential direction, T. Due to variations in growth ring orientations, a pure TL crack system was difficult to achieve and the deviation was measured to be approximately 20–30° in the TR-plane, where R denotes the radial direction. The 10 mm deep notch parallel to the grain, illustrated in Fig. 2a, was cut right prior to the tests being conducted.

The specimens were simply supported and loaded in three-point-bending, according to Fig. 2b. At one end, the specimens were placed on a steel prism resting on a steel ball and at the other on a steel prism resting on a steel cylinder, which in turn rested on ball bearings to reduce influence of friction. The span between the supports was 120 mm and the load, P, was applied at the midpoint, through a rounded cross head and a steel prism with a mass of $m_{prism} = 2.69$ g. The load was applied with a material testing system, MTS

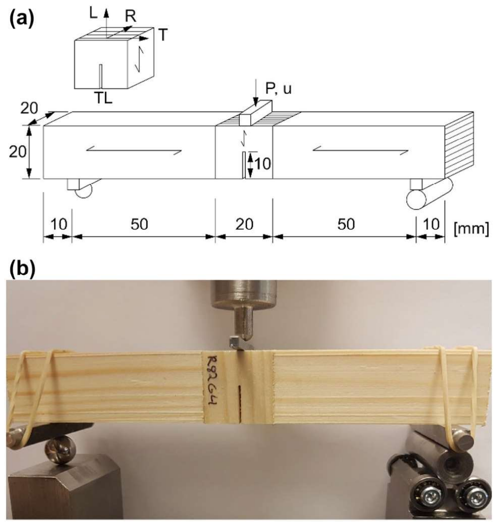

Fig. 2 SENB specimen tested in three-point-bending in the fracture energy tests, where the specimen is composed of three wood pieces glued together. **a** Specimen geometry and material orientations, where L denotes the longitudinal direction, R the radial direction and T the tangential direction of the growth ring orientation. **b** Test set-up with supports visualised

810, with displacement controlled movement of the cross head at a rate of 3 mm/min. All specimens were loaded until complete failure, recording load and mid-point displacement, the latter through the cross-head movement of the testing machine. The fracture energy was evaluated by the work done by the midpoint force and the dead weight of the specimen, divided by the fractured area, as indicated in the standard (NT BUILD 422 1993). Thus, the fracture energy perpendicular to the grain in mode I was evaluated as:

$$G_f = \frac{W + (5/6\, m_{tot} + 2\, m_{prism}) g u_0}{A_c}$$

where m_{tot} the weight of the specimen, m_{prism} the weight of the steel prism placed under the applied midpoint force, u_0 the displacement of the cross head at failure, A_c the fractured area and g the gravity acceleration. The work done by the midpoint force, W, was determined by numerical integration of the load–displacement response, using the trapezoidal method implemented in the software MATLAB.

2.3 Modulus of elasticity

The modulus of elasticity in compression parallel to the grain, E_L, was determined for 16 modified and 16 unmodified samples consisting of sapwood. The geometry of each specimen was approximately 20 mm × 20 mm × 60 mm, in the radial, tangential and longitudinal directions, respectively, as illustrated in Fig. 3a.

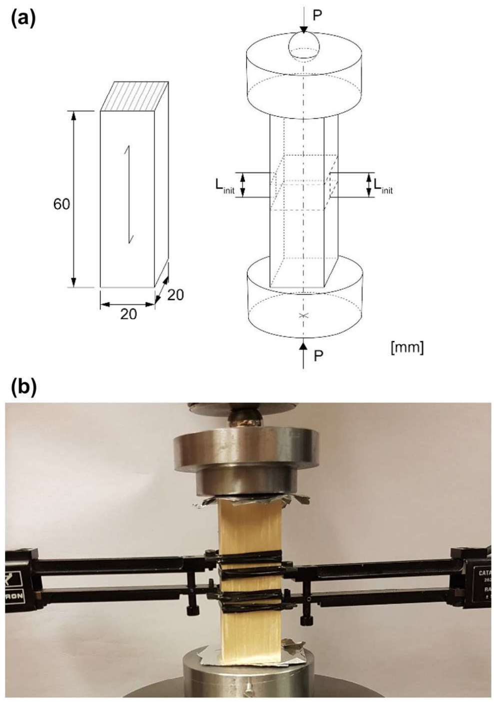

Fig. 3 Test set-up for determining the modulus of elasticity in compression along the grain. **a** Specimen geometry and location of extensometer pins, mounted on the specimen. **b** Test set-up with supports and extensometers visualised

The tests were conducted with a material testing system, MTS 810, and the specimens were loaded in the grain direction. On top of the specimen a steel cylinder was placed, with a steel ball placed in a centred cavity according to Fig. 3b. To reduce constraining shear forces, 3 layers of greased aluminium foil were placed in the interface between the specimen and the supports on both sides. The load, P, was applied with a displacement-controlled movement of the crosshead with a rate of 0.5 mm/min. The force was recorded by the testing machine, and the relative displacement by using two external extensometers, placed on opposite sides of the specimen as shown in Fig. 3. The initial distance between the extensometer pins mounted on the specimen was L_{init} = 12.5 mm.

The strain was determined based on an averaged value of the recorded relative displacements from the two extensometers and the initial length L_{init}. The stress in the longitudinal direction was evaluated by the applied force, P, divided by the cross-section area. In order to find a proper fit to recorded data, a regression line was fitted to test data corresponding to load values in the range 2 kN < P < 8 kN (corresponding to stress values of 5–20 MPa), according to the method of least squares. The slope of the regression line was used to estimate the modulus of elasticity parallel to the grain.

2.4 Tensile strength

The tensile strength perpendicular to the grain, $f_{t,90}$, was determined for 5 unmodified and 6 modified samples consisting of sapwood. The geometry of each specimen was approximately 20 mm × 20 mm × 20 mm, in the radial, tangential and longitudinal directions respectively. A cylindrical notch with a radius of 5 mm was made, as illustrated in Fig. 4a. Prior to testing the specimens, they were glued with a cyanoacrylate adhesive to steel cylinders and stored at a RH of 60% and a temperature of 20 °C until the glue had cured. The cyanoacrylate adhesive was chosen based on pre-tests to find a suitable adhesive that would provide a combination of sufficient strength yet curing fast enough to simplify specimen handling.

The steel cylinders were connected to a material testing system, MTS 810, with hinged ends to allow mounting of the specimens without introducing any constraining forces prior to testing, see Fig. 4b. The specimens were loaded at a rate of 1 mm/min until complete failure and the applied tensile load was recorded by the testing machine. The tensile strength, $f_{t,90}$, was evaluated by the maximum recorded load, P_{max}, divided by the fractured area A_c.

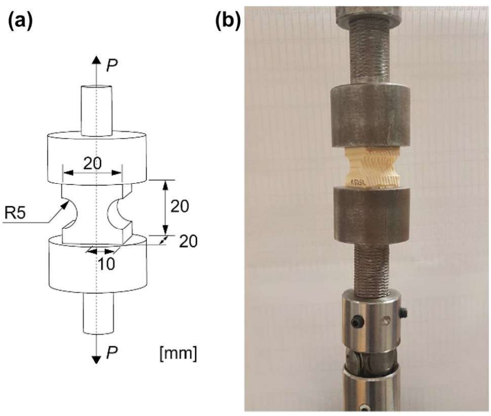

Fig. 4 Test set-up for determining the tensile strength perpendicular to the grain. **a** Specimen geometry and material orientations. **b** Test set-up with supports visualised

3 Results and discussion

3.1 Moisture content

Mean values and standard deviation of the moisture content (MC) for all examined test groups (USW; MSW; UHW; MHW) are presented in Table 1. The results clearly show a decreased moisture content for acetylated samples, which is an expected result (Rowell and Dickerson 2014). However, the measured moisture contents are slightly lower than values reported for acetylated Scots pine in previous studies (Epmeier and Kliger 2005). This is most probably attributed to a higher acetyl content of the samples investigated in this study, but it could also be a consequence of the examined samples being extremely dry prior to conditioning, or due to differences regarding acetylation techniques, climatic conditions and differences related to the origins of the material.

3.2 Fracture energy

All tests performed displayed a well-defined descending part of the load–displacement response, indicating a stable crack propagation. The work done by the midpoint force could thus be determined by numerical integration of the load–displacement response. In Fig. 5, typical load–displacement responses for unmodified and modified sapwood and heartwood are shown. Clear differences can be observed, for example, regarding the peak load, indicating a lowered fracture energy for the modified samples. Figure 6a illustrates a specimen under loading, as the crack propagates. In Fig. 6b, a typical fracture surface demonstrating a wavy pattern is shown, an attribute observed among all specimens.

Mean values and standard deviations for the fracture energy, G_f, and density, ρ, of corresponding specimens, are presented in Table 2. The difference in mean fracture energy between unmodified and modified sapwood, as well as heartwood, are presented in [J/m^2] and [%]. Compared

Table 1 Mean values of the moisture content for unmodified sapwood (USW), modified sapwood (MSW), unmodified heartwood (UHW) and modified heartwood (MHW), after conditioning at a RH of 60% and a temperature of 20 °C (numbers within brackets specify the standard deviations)

Test group	No. samples	MC [%]
USW	4	10.3 (0.3)
MSW	4	2.5 (0.2)
UHW	3	10.4 (0.6)
MHW	3	3.5 (0.5)

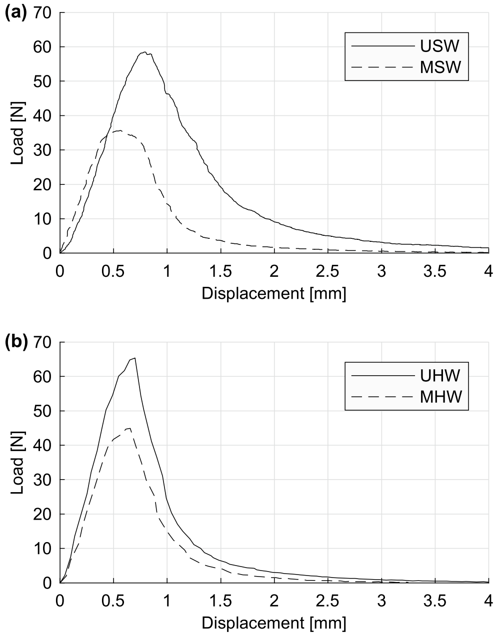

Fig. 5 Typical load–displacement responses from fracture energy tests. **a** Unmodified sapwood (USW) and modified sapwood (MSW). **b** Unmodified heartwood (UHW) and modified heartwood (MHW)

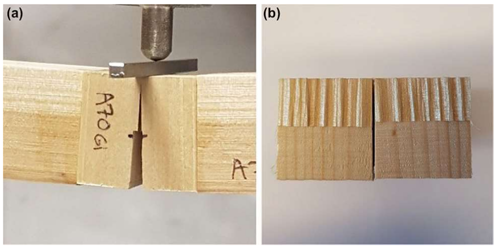

Fig. 6 Illustrations of a specimen during and after the fracture energy test. **a** Propagating crack. **b** Fracture surface

to unmodified samples, a decreased fracture energy was observed for the acetylated specimens. The mean values were 36% and 50% lower for acetylated heartwood and sapwood, respectively. The statistical significance of the difference is presented in the table through p-values and confidence intervals, according to a two-sample t-test, assuming unequal variances. There was an impact on the fracture energy at a level of significance greater than 99.9%. This observation clearly demonstrates an increased brittleness of acetylated Scots pine, which is in agreement with previous studies for another species (Reiterer and Sinn 2002). However, the observed impact appears larger in the current study. This could be due to differences in acetylation techniques, acetyl content, climatic conditions or species-specific effects (Scots pine versus spruce). It should also be emphasized that the acetylation process used in this study was optimised for radiata pine and not Scots pine.

In Fig. 7, the correlation between measured fracture energy and density are illustrated for each test group: USW; MSW; UHW; MHW. Data from all tested specimens are presented and within each plot equal markers indicate data from nominally equal specimens. A regression line is fitted to the data according to the method of least squares. A previous study conducted by Larsen and Gustafsson (1990) showed a positive correlation between the fracture energy and density for unmodified European softwoods. In the current study, the observed values of fracture energies for the unmodified specimens correspond well with the values reported by Larsen and Gustafsson (1990). In the present study, however, the correlation between the fracture energy and density was very low for same cases ($R^2=0.12$, see Fig. 7a) and the corresponding trend lines indicated in Fig. 7 cannot be considered as being statistically significant. Hence, additional testing with a wider range of densities is recommended.

Table 2 Mean values of density and fracture energy for unmodified sapwood (USW), modified sapwood (MSW), unmodified heartwood (UHW) and modified heartwood (MHW), where numbers within brackets specify the standard deviation

Test group	No. samples	ρ [kg/m^3]	G_f [J/m^2]	$\Delta\ G_f$ [J/m^2]	$\Delta\ G_f$ [%]	p-value
USW	16	475 (16)	339 (27)			
MSW	16	511 (33)	169 (17)	$-170\pm30^*$	− 50	1.5e−17
UHW	12	443 (46)	249 (23)			
MHW	11	482 (26)	158 (15)	$-91\pm31^*$	-36	4.9e−10

Differences in mean values of the fracture energy between unmodified and modified specimens, are presented in [J/m^2] and [%]. Statistical significance according to a two-sample t-test, defined by a p-value and a confidence interval

*99.9% confidence interval

3.3 Modulus of elasticity

Results for the modulus of elasticity parallel to the grain, E_L, and the density, ρ, of the corresponding samples are presented in Table 3. Mean values and standard deviations are stated, as well as the difference in mean modulus of elasticity between unmodified and modified sapwood. The statistical significance is presented by a p-value, according to a two-sample t-test with unequal variances, and a confidence interval with a significance level of 95%. Based on the statistical data, no significant impact on the modulus of elasticity could be concluded for specimens conditioned at the specified relative humidity and temperature.

The correlation between the modulus of elasticity and the density is presented in Fig. 8. The results are presented separately for unmodified and modified sapwood, and within each plot equal markers represent nominally equal specimens. The results suggest a positive correlation, i.e. an increasing modulus of elasticity for an increased density, which is expected (Kollmann 1968). However, as previously stated, statistically significant observations regarding

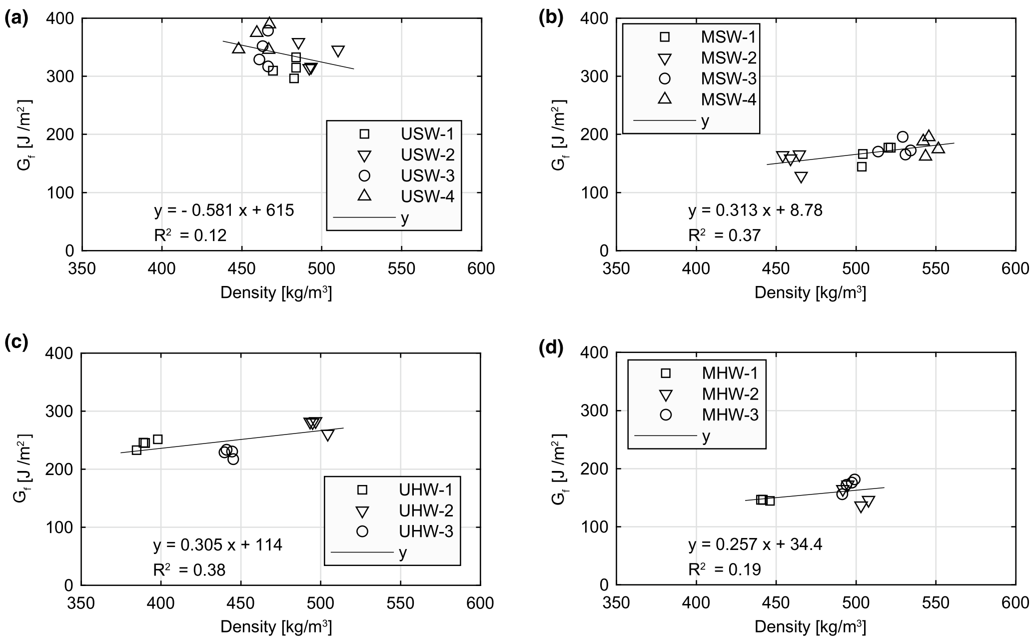

Fig. 7 Correlation between the fracture energy and the density, where nominally equal specimens are indicated with equal markers. **a** Unmodified sapwood (USW). **b** Modified sapwood (MSW). **c** Unmodified heartwood (UHW). **d** Modified heartwood (MHW)

Table 3 Mean values of density and modulus of elasticity parallel to the grain for unmodified sapwood (USW) and modified sapwood (MSW), where numbers within brackets specify the standard deviation

Test group	N.o. samples	ρ [kg/m^3]	E_L [GPa]	ΔE_L [GPa]	ΔE_L [%]	p-value
USW	16	472 (19)	12.6 (1.8)			
MSW	16	511 (35)	11.9 (0.9)	– 0.7 ± 1*	– 6	0.18

Differences in mean modulus of elasticity between unmodified and modified specimens, presented in [GPa] and [%]. Statistical significance according to a two-sample t-test, defined by a p-value and a confidence interval

*95% confidence interval

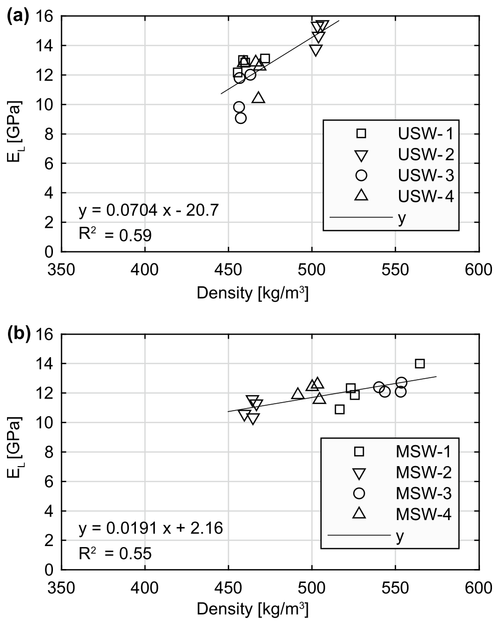

Fig. 8 Correlation between the modulus of elasticity and the density, where nominally equal specimens are indicated with equal markers. **a** Unmodified sapwood (USW). **b** Modified sapwood (MSW)

the correlation would require results from a wider range of densities and further testing is recommended.

3.4 Tensile strength

Mean values and standard deviation for the tensile strength, $f_{t,90}$, and the density, ρ, of the corresponding samples are presented in Table 4 and an example of a typical failure surface is shown in Fig. 9. The difference in mean tensile strength between unmodified and modified sapwood is presented in [MPa] and [%]. The statistical significance is defined by a p-value, according to a two-sample t-test with unequal variances, and a confidence interval with a significance level of 95%. Based on the result, no significant differences could be concluded regarding the tensile strength between unmodified and modified samples. However, for the tensile strength, it should be emphasised that these results only provide an indication of the effect of acetylation due to the limited number of samples.

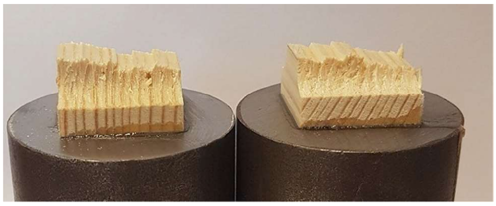

Fig. 9 A typical failure observed among the specimens in the tensile strength test

In Fig. 10, the correlation between the tensile strength and the density is shown for unmodified and modified samples consisting of sapwood. Data from all tested specimens are presented, and within each plot, equal markers indicate data from nominally equal specimens. As for the fracture energy and the modulus of elasticity, conclusions regarding the correlation of the tensile strength to the density would require examination of a wider range of densities. Thus, further testing is recommended.

3.5 Discussion

As previously mentioned, the impact of chemical modification on mechanical properties can be considered to be a consequence of changed physical properties, caused by changing the chemistry of the cell wall polymers. As stated by Bongers and Beckers (2003) as well as Larsson and Simonson (1994), changes in the mechanical properties of modified wood can be regarded as a compilation of positive effects, gained by the lower moisture content, and negative effects, impaired by having less fibres per cross-section area. For unmodified wood, a lower moisture content correlates to increased strength and stiffness (Kollmann 1968). Based on

Table 4 Mean values of density and tensile strength perpendicular to the grain for unmodified sapwood (USW) and modified sapwood (MSW), where numbers within brackets specify the standard deviation

Test group	N.o. samples	ρ [kg/m^3]	$f_{t,90}$ [MPa]	$\Delta f_{t,90}$ [MPa]	$\Delta f_{t,90}$ [%]	p-value
USW	5	484 (8)	2.7 (0.3)			
MSW	6	501 (31)	2.5 (0.2)	− 0.2 ± 0.4*	− 9	0.20

Differences in mean values of the tensile strength between unmodified and modified specimens, are presented in [MPa] and [%]. Statistical significance according to a two-sample t-test, defined by a p-value and a confidence interval. *95% confidence interval the results of the current study, no influence of acetylation on the strength nor the stiffness can be concluded. However, it should be emphasised that the current study considers samples conditioned at equal climates. Due to differences in hygroscopicity between unmodified and acetylated samples, a specific climate will result in different EMC for modified and unmodified wood. The mean moisture content for unmodified specimens was found to be 10.3–10.4%, while it was 2.5–3.5% for modified samples. Due to the strong correlation between the moisture content and the strength and stiffness for unmodified wood, the results and conclusions in this study might have been different if samples had been compared after conditioning at different climates yielding the same EMC.

In contrast to increased stiffness and strength for a decreased moisture content, studies have demonstrated that a low moisture content correlates to a decreased fracture energy (Phan et al. 2017; Reiterer and Tschegg 2002; Vasic & Stanzl-Tschegg 2007). In the current study, a significant decrease in the fracture energy was observed for the acetylated samples. To investigate why acetylation impairs the fracture energy, additional research is required. This could for instance include studies of the correlation between the fracture energy and the moisture content for modified and unmodified samples. Findings from such a study could provide an indication of whether the decreased fracture energy is merely a consequence of a drier, hence more brittle, material. Yet, considering the use of acetylated wood in structural elements, it is still of a practical importance to compare samples subjected to equal climatic conditions, i.e. temperature and relative humidity. Similarly, this study examined samples of equal dimensions. The lower fracture energy could also be an adverse effect, caused by having less fibres per cross section area. Nevertheless, for engineering practice and in design applications, the current comparison is valid since structural design is based on nominal (gross) dimensions.

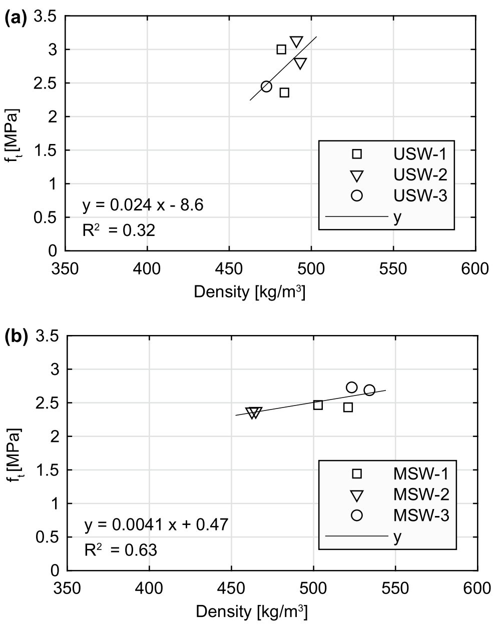

Fig. 10 Correlation between the tensile strength and the density, where nominally equal specimens are indicated with equal markers. **a** Unmodified sapwood (USW). **b** Modified sapwood (MSW)

Another possible explanation for the decreased fracture energy, could be degradation of the cell wall polymers. During the acetylation process the material is subjected to elevated temperatures (approximately 120–130 °C) at drying prior to acetylation, in the reaction with acetic anhydride as well as during the removal of the by-product acetic acid. Previous studies on thermally modified wood have indicated a significant decrease in the fracture energy (Majano-Majano et al. 2012; Reiterer and Sinn 2002). Although temperatures applied in chemical modification methods are not as high as in thermal modification methods, the cell wall polymers may still be affected. Moreover, as stated by Bongers and van Zetten (2017) and Bongers and Uphill (2019), time, temperature and pressure are key parameters to attain a consistent and uniform treatment in the process of acetylation. It is important to base these parameters on a deep knowledge of the specific wood species. Otherwise, the acetylation process might lack in uniformity, resulting in an uneven distribution of acetyl groups, and might cause internal stresses, possibly resulting in the formation of cracks. As the examined material was treated with an acetylation process optimised for radiata pine and not Scots pine, it would be of interest to examine the microstructure of the wood prior to testing, to analyse the occurrence of already existing micro-cracks that might have affected the outcome of the study.

To enable the use of acetylated young Scots pine in outdoor load-bearing applications, the increased brittleness has to be regarded in the design of mechanical joints and in structures where tensile stresses perpendicular to grain appear. The fracture energy is, for example, important for the load-bearing capacity of joints, subjected to a load at an angle to the fibre direction, and when determining edge distances to avoid brittle failure modes. Some design formulae in Eurocode 5 are based on fracture mechanics, but include assumptions regarding fracture characteristics, determined through empirical testing. Further studies are needed to determine if current design codes have to be revised, to account for the increased brittleness of modified wood.

4 Conclusion

In this study, unmodified and modified samples of Scots pine were examined. Modified samples had an acetyl content of approximately 20%, and all specimens were conditioned until equilibrium at a RH of 60% and a temperature of 20 °C. Acetylated samples demonstrated a significantly lower moisture content than unmodified samples. Significant differences were also observed regarding the fracture energy, where the mean value decreased with 36% and 50% for acetylated heartwood and sapwood, respectively. No significant effects of the acetylation regarding tensile strength perpendicular to the grain, nor modulus of elasticity parallel to the grain, could be concluded. The observations demonstrate an increased brittleness for acetylated Scots pine. This fact is important to regard in the design of mechanical joints as well as in structural elements where tensile stresses perpendicular to grain appear. Based on the knowledge gained, further studies will be conducted regarding structural applications to determine whether current design codes have to be revised, in order to account for the increased brittleness of acetylated Scots pine.

To further investigate the cause of the decreased fracture energy, both modified and unmodified specimens conditioned at a range of moisture contents should be examined. By doing so, the effect of MC on the fracture energy can be separated from other effects, such as the changed chemistry and the amount of wood fibres. Furthermore, it would be of interest to investigate the microstructure of the modified wood, to study the presence of micro-cracks and determine whether there is a degradation of the cell wall polymers.

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Compliance with ethical standards

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

References

Bongers F, Beckers E (2003) Mechanical properties of acetylated solid wood treated on pilot plant scale. In: Proceedings of the first european conference on wood modification, Ghent, Belgium

Bongers F, Uphill SJ (2019) The potential of wood acetylation. In: 7th-international scientific conference on hardwood processing. Delft, The Netherlands

Bongers F, van Zetten J (2017) Consistency of performance of acetylated wood. The international research group on wood protection, IRG-WP 17- 20608

Brelid PL (2013) Benchmarking and state of the art for modified wood. SP Technical Research Institute of Sweden, Stockholm

Cabrero JM, Honfi D, Jockwer R, Yurrita M (2019) A probabilistic study of brittle failure in dowel-type timber connections with steel plates loaded parallel to the grain. Wood Mat Sci Eng 14(5):298–311

Dourado N, de Moura MSFS, Morais J (2011) A numerical study on the SEN-TPB test applied to mode I wood fracture characterization. Int J Solids Struct 48(2):234–242

Dourado N, de Moura MSFS, Morel S, Morais J (2015) Wood fracture characterization under mode I loading using the three-point-bending test. Experimental investigation of Picea abies L. Int J Fract 194:1–9

Dreher WA, Goldstein IS, Cramer GR (1964) Mechanical properties of acetylated wood. Forest Products Journal 14:66–68

Ehlbeck J, Görlacher R (2017) E11 Joints loaded perpendicular to the grain. In: Blass HJ, Sandhaas C (eds) Timber engineering—principles for design. KIT Scientific Publishing, Karlsruhe, pp 441–457

Epmeier H, Kliger R (2005) Experimental study of material properties of modified Scots pine. Holz Roh- Werkst 63:430–436

Fuchs W (1928) Genuine lignin. I. Acetylation of pine wood. Ber Dtsch Chem Ges 61:948–951

Giotra K (2014) Process for wood acetylation and product thereof, *European Patent No. 2818287A1*. European Patent Office

Goldstein IS, Jeroski EB, Nielson JF, Weaver JW (1961) Acetylation of wood in lumber thickness. Forest Products J 11:363–370

Gustafsson PJ (2003) Fracture perpendicular to grain—structural applications. In: Thelandersson S, Larsen HJ (eds) Timber engineering. Wiley, Chichester, pp 114–115

Jensen JL, Quenneville P (2011) Experimental investigations on row shear and splitting in bolted connections. Constr Build Mater 25(5):2420–2425

Kollmann FFP (1968) Mechanics and rheology of wood. In: Kollmann FFP, Côté WA (eds) Principles of wood science and technology. Springer, Berlin, Heidelberg, pp 292–419

Larsen HJ, Gustafsson PJ (1990) The fracture energy of wood in tension perpendicular to the grain: results from a joint testing project. In: International council for research and innovation in building and construction, working commission CIB-W18A, proceedings Meeting 23, Paper CIB-W18A/23–19–2, Lisbon, Portugal

Larsson P, Simonson R (1994) A study of strength, hardness and deformation of acetylated Scandinavian softwoods. Holz Roh- Werkst 52:83–86

Majano-Majano A, Hughes M, Fernandez-Cabo JL (2012) The fracture toughness and properties of thermally modified beech and ash at different moisture contents. Wood Sci Technol 46:5–21

Morel S, Dourado N, Valentin G, Morais J (2005) Wood: a quasibrittle material R-curve behavior and peak load evaluation. Int J Fract 131(4):385–400 de Moura MSFS, Dourado N, Morais J (2010) Crack equivalent based method applied to wood fracture characterization using the single edge notched-three point bending test. Eng Fract Mech 77(3):510–520

NT BUILD 422 (1993) Wood: Fracture energy in tension perpendicular to the grain. Nordtest Method 11/1993

Phan NA, Chaplain M, Morel S, Coureau JL (2017) Influence of moisture content on mode I fracture process of *Pinus pinaster*: evolution of micro-cracking and crack-bridging energies highlighted by bilinear softening in cohesive zone model. Wood Sci Technol 51(5):1051–1066

Reiterer A, Sinn G (2002) Fracture behaviour of modified spruce wood: a study using linear and non linear fracture mechanics. Holzforschung 56:191–198

Reiterer A, Tschegg S (2002) The influence of moisture content on the mode I fracture behaviour of sprucewood. J Mater Sci 37(20):4487–4491

Rowell RM (1983) Chemical Modification of Wood. Forest Products Abstracts 6(12):363–382

Rowell RM (1996) Physical and mechanical properties of chemically modified wood. Chem Modification Lignocellulosic Materials 1:295–310

Rowell RM (2006) Chemical modification of wood: a short review. Wood Mat Sci Eng 1:29–33

Rowell RM, Dickerson JP (2014) Acetylation of wood. In: Schultz TP, Goodell B, Nicholas DD (eds) Deterioration and protection of sustainable biomaterials. American Chemical Society, Washington, pp 301–327

Schwanninger M, Stefke B, Hinterstoisser B (2011) Qualitative assessment of acetylated wood with infrared spectroscopic methods. J Near Infrared Spectrosc 19(5):349–357

Sjödin J, Serrano E (2008) A numerical study of methods to predict the capacity of multiple steel-timber dowel joints. Holz Roh- Werkst 66:447–454

Vasic S, Stanzl-Tschegg S (2007) Experimental and numerical investigation of wood fracture mechanisms at different humidity levels. Holzforschung 61(4):367–374

Paper B

Moisture-dependency of the fracture energy of wood: A comparison of unmodified and acetylated Scots pine and birch

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Abstract

The moisture-dependency of the fracture energy for unmodified and acetylated Scots pine (*Pinus sylvestris* L.) and birch (*Betula pendula* Roth) has been investigated. Specimens were conditioned at relative humidity levels of 20%, 75%, and 97%, as well as dry and water-saturated. At moisture contents below 15%, the fracture energy increased with increasing moisture content for both unmodified and acetylated wood, while it decreased for untreated wood at higher moisture contents. A significant difference in moisture-dependency was found, indicating higher fracture energy for unmodified wood compared to acetylated wood at similar moisture contents. Additionally, to assess the impact of the increased brittleness for structural applications, the fracture energy was compared at equal relative humidity levels. The largest difference was seen at 75% relative humidity with approximately 50% lower fracture energy for acetylated wood. No significant differences were found for water-saturated samples. The moisture-dependency of the fracture energy, combined with the reduced hygroscopicity of acetylated wood, is suggested to be one, but not the only, contributing factor to the lower fracture energy of acetylated wood compared to unmodified wood at equal humidity levels. These observations have importance for structural design since design codes often assess material parameters based on ambient humidity.

1. Introduction

In order to reach milestone targets in mitigating the climate change, many operators within the building sector are exploring possibilities to increase the use of timber in load-bearing structures. An increased use of wood in outdoor load-bearing structures would open possibilities for new architectural expressions. This should, in turn, increase the awareness about those possibilities and about the environmental and climatic benefits associated with the use of wood in constructions. However, wood used in outdoor conditions must be protected from moisture to avoid excessive swelling and shrinking, as well as fungal degradation. To overcome these drawbacks, many different wood modification methods have been studied. These methods typically aim at modifying the physical properties of the wood to achieve a more hydrophobic material, without introducing harmful preservative substances (Rowell 2006). One promising method is acetylation, which is based on a chemical reaction between acetic anhydride and the wood polymers, resulting in the esterification of accessible hydroxyl groups in the cell wall (Rowell 1983). The resulting change of the chemical constitution of the cell wall affects most physical attributes of the material: Acetylated wood exhibits a decreased equilibrium moisture content, a lower maximum cell wall moisture content (Rowell 2006), and due to bulking of the cell wall, it exhibits less fibres per cross section area compared to its unmodified state (Rowell 1996).

As reported by e.g. (Brelid 2013; Rowell 1983, 2006), acetylation indeed results in increased durability and improved dimensional stability. Today, acetylated wood commercially available is made from wood imported to Europe (*Pinus radiata* D. Don) and it is largely limited to non-structural applications, such as decking, furniture and facades. To reduce the climatic impact of such products, raw materials readily available in Europe could be used instead, if technically possible. Demonstrating the feasibility of using European raw materials, would also increase the economic incentive for the European forest-based industry to promote the use of acetylated wood. To make use of acetylated wood in load-bearing structures, the impact on the mechanical properties of the acetylation process must be well understood and quantified. Previous studies have investigated bending stiffness and strength of acetylated wood

(Bongers and Beckers 2003; Dreher et al. 1964; Epmeier and Kliger 2005; Larsson and Simonson 1994) and results have also been reported on the impact of acetylation on compressive strength (Bongers and Beckers 2003; Dreher et al. 1964; Goldstein et al. 1961), hardness (Bongers and Beckers 2003; Dreher et al. 1964), shear strength (Dreher et al. 1964), and impact strength (Bongers and Beckers 2003; Goldstein et al. 1961).

One identified challenge is that acetylation seems to increase the brittleness of the material (Forsman et al. 2020; Lai and Plönning 2019; Reiterer and Sinn 2002). This finding is important to verify and quantify prior to large-scale use of acetylated wood in load-bearing structures, since the occurrence of knots, holes, notches, moisture gradients etc., can induce large stress concentrations, which may lead to crack initiation and propagation (Gustafsson 2003). Based on the limited number of studies on fracture characteristics of acetylated wood, the loss in mode I fracture energy has been estimated to be approximately 20%–50% (Forsman et al. 2020; Lai and Plönning 2019; Reiterer and Sinn 2002). However, previous studies on the fracture characteristics have only considered unmodified and acetylated samples conditioned at equal climatic conditions, i.e. at equal temperature and in equilibrium with the same relative humidity (RH). Due to the decreased hygroscopicity of acetylated wood, this means that fracture characteristics of acetylated and unmodified samples have so far only been compared at unequal moisture contents. Thus, it is not yet known whether the increased brittleness of acetylated wood is simply an effect of the lower moisture content.

In this study, the fracture energy of unmodified and acetylated Scots pine and birch conditioned to equilibrium at various RH levels has been investigated. The results were used to examine the correlation between the moisture content and the fracture energy for unmodified and acetylated wood. The wider range of moisture contents/RH levels examined herein as compared to previous studies, makes it possible to estimate the significance of the increased brittleness of acetylated wood for moisture conditions relevant in the design of load-bearing structures. The wood materials examined in this study have today only limited or no use for structural purposes outdoors, due to poor durability and dimensional stability. By acetylation, it is possible to increase both durability and dimensional stability. However, research on the impact of the acetylation process on fracture characteristics of the material is essential before its use in outdoor load-bearing applications.

2. Materials and methods

2.1 Wood materials

The wood materials used were Scots pine (*Pinus sylvestris* L.) and birch (*Betula pendula* Roth). The pine was provided by a sawmill in Finland, Isojoen Saha. It consisted of small-dimension sawn timber from thinning's, aged 30–40 years in a close-by area, well-known for its fast-growing pine. The birch originated from Sweden and was provided by Vanhälls Såg AB. For birch, matched boards were investigated, i.e. one long board was split in two parts, where one part later was acetylated and the other kept unmodified. Hence, the unmodified and the modified samples of the birch had similarities in origins, density, width of growth rings, growth ring orientations etc. For the Scots pine, the unmodified and the acetylated samples originated from the same batch but not exclusively the same board.

2.2 Acetylation procedure

The modification of the acetylated boards was performed in a proprietary industrial scale process, at the Accsys Group's acetylation plant in Arnhem, the Netherlands. The standard process used in the commercial production process of Accoya radiata pine at Accsys Technologies was applied (European Patent No. 2818287A1, 2014). This process involves a chemical reaction between acetic anhydride and wood polymers at elevated temperatures of approximately 120–130ºC, without the use of catalysts. It should be noted that the process parameters were not optimized neither for Scots pine nor birch, i.e. no adjustments were made regarding time, temperature, pressure or concentration levels. The modified boards were analysed for acetyl content using near infrared spectroscopy, a method described by Schwanninger et al. (2011). All the examined acetylated boards demonstrated a weight percentage gain (WPG) above 20%. The oven-dry density of the unmodified and the acetylated spec-

Table 1: *The mean value of the oven-dry density determined after drying at a temperature of 105ºC. The numbers within brackets specify the standard deviation.*

Species	Treatment	Density, ρ (kg m^{-3})
Pine	Unmodified	406 (5)
	Acetylated	532 (12)
Birch	Unmodified	647 (17)
	Acetylated	746 (10)

imens, determined after drying at 105ºC, is presented in Table 1.

2.3 Sorption isotherms

Absorption isotherms at 20°C were determined in a sorption balance (DVS Advantage, Surface Measurement Systems, Ltd., London, UK). A sorption balance monitors the mass of a specimen (resolution 0.1 µg) while the RH is incrementally changed in pre-programmed steps, see e.g. Williams (1995). Scots pine, acetylated Scots pine, birch and acetylated birch were cut to thin small pieces using a razor blade. The total sample mass for each wood type was about 10 mg. The sample was placed in the sample pan and dried at 0% RH/20°C for 24 h. The sample was then equilibrated at the following RH levels: 20%, 40%, 60%, 80% and 95%. The equilibration criterion used at a certain relative humidity step can be expressed either as a specific time or as a criterion based on the rate of change of mass with respect to time, i.e. a dm/dt-criterion. Due to uncertainties related to the use of dm/dt-criteria (Glass et al. 2017, 2018) a time criterion was chosen in the present study. The time at each RH level was 12 h, except at 95% RH where the time was set to 24h. The sample was then dried at 60°C for 8 h using the pre-heater in the instrument, followed by a 2 h thermal stabilisation period at 20°C before the dry mass was determined. The equilibrium moisture content at each RH level was then evaluated as mass of water, i.e. the total mass at equilibrium minus the dry mass, divided by the dry mass of the wood. For the acetylated specimens, the moisture contents were corrected for the increase in dry mass obtained by the acetylation process, i.e. the moisture content was determined as (Thybring 2013):

$$u_0 = u_{mod}(1 + R_{mod}) \tag{1}$$

where u_0 (%) is the moisture content based on the dry mass before acetylation, u_{mod} is the moisture content based on the dry mass after acetylation, and R_{mod} is the relative mass increase due to the acetylation procedure. For the untreated wood, $R_{mod} = 0$.

2.4 Sample preparation

From each board consisting of either Scots pine, acetylated Scots pine, birch or acetylated birch, specimens sized 20 mm × 20 mm × 20 mm were extracted according to the pattern shown in Figure 1. To avoid influence of heartwood and juvenile wood, two sticks were extracted from the outer part of the board, one from each side of the pith (Figure 1a). In the lengthwise direction of each stick, nominally equal samples, replicates (Figure 1b), were ex-

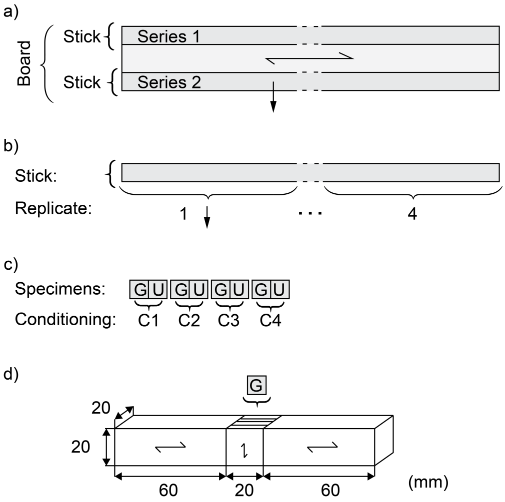

Figure 1: *The pattern used for extracting specimens from each board: Two sticks extracted from each side of the pith of the board (a); Specimens extracted for each replicate, i.e. nominally equal samples (b), and conditioning (c), where G denotes specimens for the fracture energy test and U specimens for the density and the moisture content; Specimens for the fracture energy test glued together with two more wood pieces (d).*

tracted for conditioning to different RH levels (Figure 1c). For each specimen intended for fracture energy testing (denoted G), one adjacent specimen was extracted for which density and moisture content were determined (denoted U). The extracted specimens (G) were glued with polyvinyl acetate (Dana Lim, Wood Glue D3 Outdoor 430) to two wood pieces according to Figure 1d.

2.5 Sample conditioning

Prior to conditioning, all specimens were dried at 60°C in order to ensure conditioning to the absorption isotherm and to reduce the influence of hysteresis. Specimens were conditioned at five RH levels, C1—C5 (Table 2). The specimens conditioned at C1 were oven-dried at 60°C for 7 days, and then placed in a desiccator containing molecular sieve (0.4 nm, Merck KGaA, Germany) to remain dry until tested. The specimens in conditions C2–C4 were conditioned using saturated salt-solutions in sealed boxes for 60 days, kept at a temperature of 20ºC while measuring RH to ensure stable humidity levels over time. For condition C2, sodium hydroxide (NaOH) was used, with an expected RH level of 9% (Greenspan 1977). A higher RH was, however, noted (approximately 20%), which was most likely attributed to that the solution was not fully saturated. For conditions C3 and C4, sodium chloride (NaCl) and potassium sul-

__Table 2:__ The number of specimens in the fracture energy test for condition C1-–C5 and each test group. The numbers within brackets specify the original number assigned for each test group/condition, noted where specimens were excluded due to sources of error prior or during testing.

Species	**Treatment**	**C1 OD**	**C2 RH 20%**	**C3 RH 75%**	**C4 RH 97%**	**C5 WS**
Pine	Unmodified	4	8	7 (8)	6 (8)	4
	Acetylated	4	8	8	8	4
Birch	Unmodified	4	8	8	8	4
	Acetylated	4	8	7 (8)	8	4

phate (K_2SO_4) were used, with expected RH levels of 75% and 97% respectively (Greenspan 1977). These levels were confirmed by measurements and remained stable over time. For condition C5, the specimens were vacuum-saturated to achieve a well-defined, fully-saturated state. The water saturation was performed by placing the specimens in vacuum (<1 mbar) in a glass desiccator for 1 h, deionized water was then added while running the vacuum pump, and finally atmospheric pressure was re-established. The specimens were then kept in water for 8 days before the fracture energy tests were performed.

The number of replicates for each condition and test group, i.e. series 1 and series 2 (Figure 1a) combined, is presented in Table 2, where specimens excluded due to sources of error prior or during test are noted. The number of specimens assigned to conditions C1 and C5 was lower due to an in-process decision to use some of the specimens originally assigned to condition C1, to also include water-saturated samples, C5. Moisture contents were determined by the oven-dry method and corrected for the increase in dry mass for acetylated wood (Eq. 1). For the water-saturated specimens (C5), an estimation of the cell wall moisture content was made since the fracture energy presumably is affected by the cell wall moisture content rather than the total moisture content. This estimation was performed as follows. For the unmodified wood, the volume of the voids outside of the cell walls was calculated from the measured dry bulk densities of the untreated wood (Table 1) and literature values of cell wall densities for the two wood species (Plötze and Niemz 2011). The amount of water in the cell walls in the saturated state was then determined by subtracting the amount of water in voids outside of cell walls from the total amount of water at saturation. In Thybring (2013), the relation between the WPG obtained by modification and the moisture exclusion efficiency is shown for cell wall bulking modifications. Based on this relation, and the estimated cell wall moisture content of the untreated specimens, the cell wall moisture content in the saturated state for the acetylated wood was determined. This methodology gave cell wall moisture contents for untreated Scots pine well in agreement with values for the same wood species measured using Low Field Nuclear Magnetic Resonance (Telkki et al. 2013). Also, the values estimated for acetylated Scots pine were in the same range as cell wall moisture contents for acetylated radiata pine in Beck et al. (2018).

2.6 Fracture energy tests

The fracture energy is the energy dissipated in the fracture process zone, from crack initiation to creation of traction-free crack surfaces. It is the energy needed to produce a unit area of traction-free crack, measured in (J m^{-2}) (Gustafsson 2003). In this study, the fracture energy was determined according to the standard NT BUILD 422 (Nordtest method 1993), i.e. single-edge notched bend (SENB) specimens subjected to three-point bending were used to determine the mode I fracture energy in tension perpendicular to the grain. The main reason for choosing this test method and the related evaluation methods, was its simplicity: it only requires the evaluation of the energy put into the system by the load applied, and the only assumption made is the assumption of a negligible influence of plastic dissipation. The influence of plastic dissipation on the results can, as stated by Gustafsson (2003), be considered negligible for small specimens, like the ones used in this study. The current study thus included measurements of fracture energy but not measurements of the fracture process itself in terms of e.g. crack opening displacement (COD) or tracking of the crack formation and propagation. Such measurements could be an interesting further development for future studies using digital image correlation techniques (DIC). Such characterisation has been done for wood adhesive bonds (Serrano and Enquist, 2005) and in more recent work for wood fracture by Dourado et al. (2015), Majano-Majano et al. (2019) and Ostapska and Malo (2020).

Prior to the test, a 10 mm notch was made in the fracture energy specimen (Figure 2b). For specimens made from pine, a rectangular notch was made, according to the standard NT BUILD 422

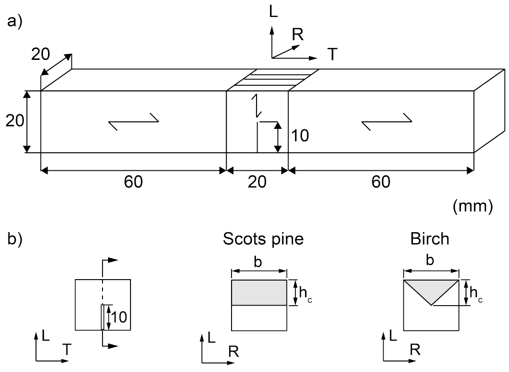

Figure 2: *Geometry and orientations of the SENB-specimen (a) with notch geometries shown by an intersection (b) for Scots pine (rectangular ligament) and birch (triangular ligament). The coordinate systems are defined by the longitudinal (L), tangential (T) and radial (R) directions.*

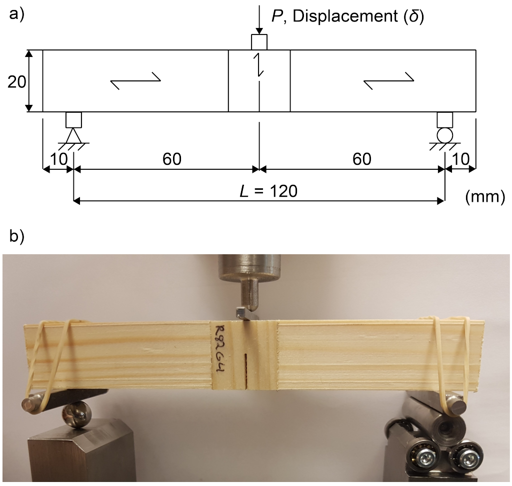

Figure 3: *Test-setup and geometry for the fracture energy test (a), where the SENB-specimen was simply supported and loaded in three-point bending (b).*

(Nordtest method 1993). For birch, an adjustment of the standard procedure was made regarding the notch geometry and a triangular ligament was realised instead of the rectangular one described in the standard. This adjustment was made in order to obtain a stable test performance, which would otherwise be difficult to obtain as found in (Lai and Plönning 2019). The central piece was oriented aiming at a TL-crack propagation system, i.e. with the crack propagating in the longitudinal direction (L) and the normal to the fracture surface in the tangential direction (T) (Figure 2a). The deviation from a pure TL-crack propagation system was measured as 20-–30 degrees in the RT-plane, where R denotes the radial direction and T the tangential direction.

As shown in Figure 3, the SENB specimens were simply supported and the span between the supports was 120 mm. At one end, the specimen was placed on a steel prism resting on a steel ball, and at the other end on a steel prism resting on a steel cylinder, which in turn rested on ball bearings. The specimens were loaded in three-point bending by a load, P, applied at the midpoint with a rounded loading-nose, through a steel prism with a mass of 2.69 g. The tests were performed with a Material Testing System (MTS-810), and the load was applied with displacement-controlled movement of the cross head at a rate of 3 mm min^{-1}. All specimens were loaded until complete failure, and the load was recorded by a load cell (MTS 661.11B-02) and the mid-point displacement via the cross-head displacement of the testing system, through the build-in transducer of the machine. The fracture energy was evaluated by calculating the work done by the midpoint force and the dead-weight of the specimen, and divide this work by the fractured area, as described in the standard NT BUILD 422 (Nordtest method 1993). The work done was determined by numerical integration of the load-displacement response, using the trapezoidal method, trapz, implemented in the software MATLAB (MATLAB R2017b, The MathWorks Inc., Natick, MA, US).

2.7 Evaluation of stable responses

According to the standard NT BUILD 422 (Nordtest method 1993), only stable responses should be evaluated. Stable responses refer to load-displacement responses for which it is possible to record the softening branch of the load versus deformation curve. However, no criterion for classification of the response as being stable or unstable is given in the standard. To evaluate the influence of possible unstable responses, the maximum relative load drop between two consecutive sampling points (relative to the maximum load recorded) was chosen to represent the degree of stability of the response. Thus, a restriction criterion, LC (%), was introduced:

$$LC = |P_i - P_{i+1}|/P_{max} \times 100 \tag{2}$$

with definitions according to Figure 4. In this study, the influence of varying the allowable level of LC was investigated, with the aim of verifying the sensibility of the fracture energy evaluation method to minor instabilities. Four different LC-levels were investigated, representing allowing any, 15%, 10% and 5% load drop, respectively.

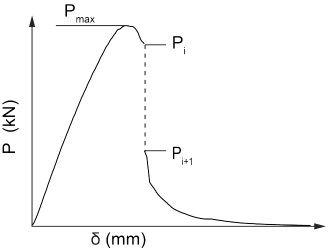

Figure 4: *Definition of the maximum load (P_{max}), and the load in two consecutive sampling points (P_i and P_{i+1}), used to determine the criterion LC (Eq. 2), which defines instability of the load-displacement response.*

3. Results and discussion

3.1 Moisture content and sorption isotherms

As wood is a hygroscopic material, its moisture content will depend on ambient RH and temperature. To establish this relation for the materials investigated, sorption isotherms were evaluated for each test group. Figure 5 shows the sorption isotherms for the unmodified and the acetylated Scots pine and birch. As expected (Rowell and Dickerson 2014), a decreased hygroscopicity was observed for both wood species when acetylated. The moisture contents for acetylated specimens did not exceed 11% at RH levels up to 97%.

Moisture contents for the specimens used in the fracture energy test (evaluated for samples denoted U according to Figure 1) were determined after conditioning at C1—C5. Mean values of the moisture content for C1—C4 are shown in Figure 5, together with the absorption isotherms obtained by sorption balance measurements. As the expected RH levels may lack in accuracy due to various sources of error, and since a long equilibration time is needed for large specimens in climate boxes, the moisture contents for the specimens used in the fracture energy test were compared to the measured absorption isotherms. Figure 5 shows that the moisture contents for the larger specimens conditioned in climate boxes were in general well in line with the sorption isotherms determined for smaller samples. Thus, the 60-days equilibration time was indeed enough to reach equilibrium at each RH level. The results in Figure 5 also confirm that the RH level for condition C2 was higher than intended (20% instead of 9%). It can also be observed that the moisture content for the unmodified specimens tested at condition C1 deviated from zero. To remove all moisture, higher temperatures are required, but the temperature of 60ºC was chosen not to interfere with the binding of acetyl groups and wood polymers.

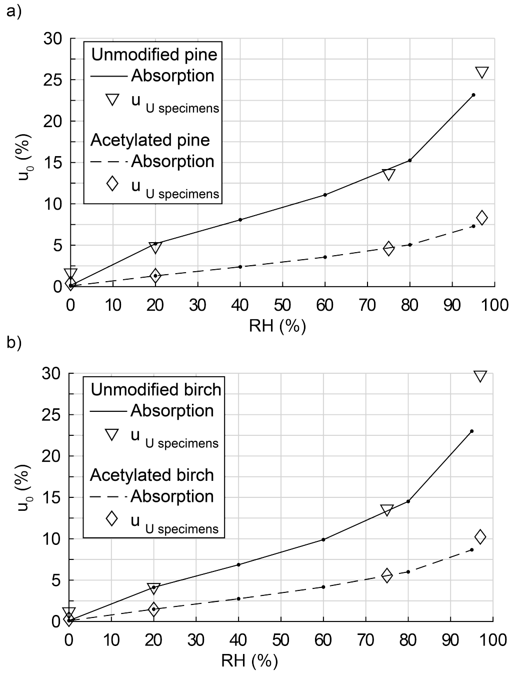

Figure 5: *Sorption isotherms for the unmodified and the acetylated Scots pine (a) and birch (b), measured at a temperature of 20ºC in a sorption balance. Mean values of the measured moisture contents for the specimens (denoted U) conditioned at condition C1—C4, are marked with triangles (unmodified wood) and squares (acetylated wood). The moisture contents for acetylated wood is corrected according to Eq. 1.*

3.2 Typical load-displacement responses

According to the method presented, load and displacement were recorded during the fracture energy tests. To account for variations in notch geometries, and to make a comparison between the responses easier, the recorded load values, P, were converted to nominal stress values, σ_f, by:

$$\sigma_f = PL/4W \quad (3)$$

$$W = (bh_c^2)/6 \quad (4)$$

$$W = (bh_c^2)/24 \quad (5)$$

where W is the elastic section modulus (Eq. 4 for a rectangular cross section, Eq. 5 for a triangular cross section), h_c the height of the fractured area (Figure 2b), b the width of the specimen (Figure 2b), and L the span width between the supports (Figure 3). Typical stress-displacement responses, based on this conversion and for each condition, are shown in Figure 6. Two observations were made for both wood species: (1) No impact of the RH

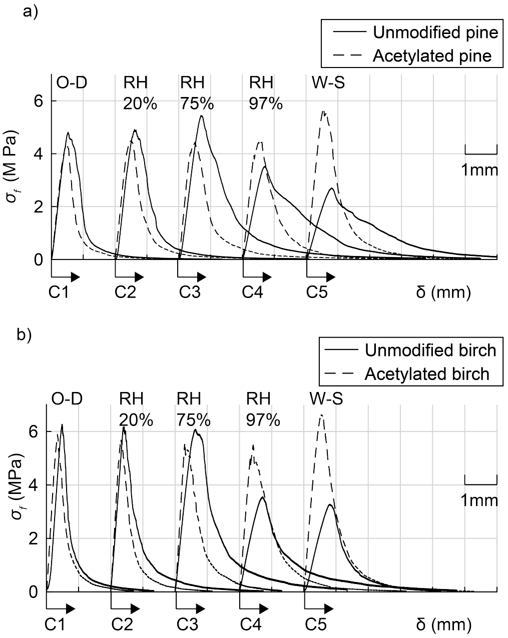

Figure 6: *Typical stress (σ_f) versus displacement (δ) responses for unmodified and acetylated Scots pine (a) and birch (b), after conditioning at C1–C5. The responses for each condition are presented with a 2 mm offset from each other along the x-axis.*

on the initial stiffness was seen for the acetylated samples, whereas a decreased stiffness was observed for the unmodified samples at RH levels exceeding 75%. (2) For an increased RH level above 75%, the maximum stress before softening decreased for the unmodified samples while no impact was noticed for the acetylated samples. These observations were expected as lower moisture contents correlate to higher stiffness and strength (Kollmann 1968). That the initial stiffness and failure strength for the acetylated samples were not affected in the same manner can possibly be explained by the decreased hygroscopicity of acetylated wood.

3.3 Evaluation of stable responses

Depending on the restriction criteria applied to discard unstable test performance results (the relative load drop, LC (Eq. 2)), mean values and standard deviations of the fracture energy may vary. Figure 7 shows the fracture energy statistics for each RH level considering different LC:s (All, 15%, 10% and 5%). As can be observed, the influence of excluding tests with unstable responses was found to be of minor importance. The overall tendencies observed (influence of RH and interrelation between unmodified and acetylated specimens) were not affected. In addition, no major impact was found regarding the

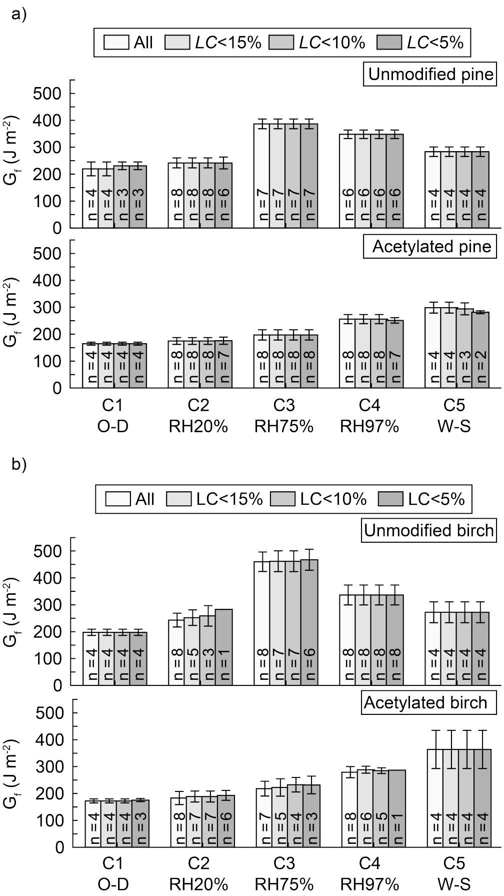

Figure 7: *The mean values and the standard deviations of the fracture energy (G_f) for unmodified and acetylated Scots pine (a) and birch (b), depending on applied restriction criterion, LC (Eq. 2). The number of included responses, n, for the different criteria are denoted in the corresponding bar.*

mean values within the test groups. Thus, it can be concluded that including all load-displacement responses in the estimation of the fracture energy was reasonable for this study, and all the following results will be based on the number of specimens given in Table 2, i.e. criterion "All".

3.4 Fracture energy versus moisture content

Table 3 presents the fracture energy, G_f, and the moisture content, u_0 (Eq. 1), for RH levels C1-–C5. Note that for the water-saturated samples (C5) an estimation of the cell wall moisture content, u_{CW}, is presented. As previously noted by several researchers (Bongers and Beckers 2003; Larsson and Simonson 1994) the impact of the acetylation process on the mechanical properties of wood can be regarded as a consequence of

Table 3: *The mean values of the fracture energy (G_f) and the moisture content (u_0) for each test group/condition. The numbers within brackets specify the standard deviations. Note that for C5, an estimation of the cell wall moisture content (u_{cw}) is included, and the standard deviation given here is a result of the spread in wood density.*

Species	**Treatment**	**C1**		**C2**		**C3**		**C4**		**C5**		
		O-D		**RH 20%**		**RH 75%**		**RH 97%**		**W-S**		
		G_f	u_0	G_f	u_0	G_f	u_0	G_f	u_0	G_f	u_0	u_{cw}
		(J m^{-2})	(%)	(J m^{-2})	(%)	(J m^{-2})	(%)	(J m^{-2})	(%)	(J m^{-2})	(%)	(%)
Pine	Unmodified	220	1.7	242	4.9	387	13.7	348	26.1	283	216.5	37.4
		(26)	(0.1)	(19)	(0.0)	(18)	(0.1)	(16)	(0.2)	(18)	(3.5)	(3.1)
	Acetylated	165	0.4	175	1.3	197	4.6	256	8.3	299	144.5	20.3
		(5)	(0.0)	(13)	(0.1)	(19)	(0.0)	(17)	(0.1)	(20)	(7.9)	(1.7)
Birch	Unmodified	198	1.2	243	4.1	460	13.6	336	29.8	272	118.9	30.9
		(12)	(0.0)	(26)	(0.0)	(36)	(0.1)	(37)	(1.6)	(39)	(1.9)	(4.2)
	Acetylated	172	0.3	183	1.5	218	5.6	279	10.2	364	82.2	17.3
		(8)	(0.0)	(25)	(0.1)	(28)	(0.1)	(21)	(0.2)	(71)	(2.6)	(2.3)

the altered attributes (increased density, lower moisture content, less fibres per cross-section area). To visualize a possible effect of the lower moisture content, the fracture energy is plotted versus the moisture content in Figure 8. For both wood species, the following could be concluded: For unmodified wood, an increased moisture content resulted in an increased fracture energy for moisture contents up to approximately 15%, while a decreased fracture energy was observed for moisture contents exceeding this value. For acetylated wood, an increasing fracture energy was found for all the included moisture contents.

Thus, a peak (local maximum) in fracture energy was observed for the unmodified wood. Due to the limited number of data points (number of climates used for conditioning), it was not possible to draw any precise conclusions about the exact location of the peak. However, the results indicated this peak to be located at approximately 12-–18% moisture content. This observation aligned with previous studies for a TL-oriented crack propagation system, where a similar behaviour was detected for untreated beech and ash at moisture contents around 13% (Majano-Majano et al. 2012) and for red pine around 18% moisture content (Smith and Chui 1994). Amorphous polymers undergo softening, i.e. they go from a glassy to a rubbery state when the temperature changes. The temperature at which this occurs is called the glass transition temperature. The transition from a glassy to a rubbery state is, however, not only dependent on the temperature, it is also moisture dependent. For hemicellulose, which is the most hygroscopic polymer in wood, glass transition occurs in the region 60% — 90% RH at room temperature, which corresponds to moisture contents between 10% and 20% (Engelund et al. 2013). The peak in fracture energy observed for moisture contents around 12—18% could thus be attributed to this transition. Worth noting is that in previous studies, the observed peak has only been noted for TL-oriented crack propagation systems, and the moisture-dependency of fracture energy for RL-oriented crack propagation systems has been suggested to be monotonic (Majano-Majano et al. 2012; Reiterer and Tschegg 2002). For the acetylated wood, no peak was identified in the data set. This could either be because it occurs at a moisture content where no data were obtained in the present study (between data points), or, because acetylation increases the glass transition temperature. It has, in previous studies, been speculated that acetylation may change at which moisture content/humidity level glass transition occurs at room temperature (Hunt et al. 2018; Zelinka et al. 2016).

To evaluate differences in the moisture-dependency of the fracture energy between unmodified and acetylated wood, data need to be compared within a relevant interval of moisture contents, i.e. an interval for which data is available for acetylated

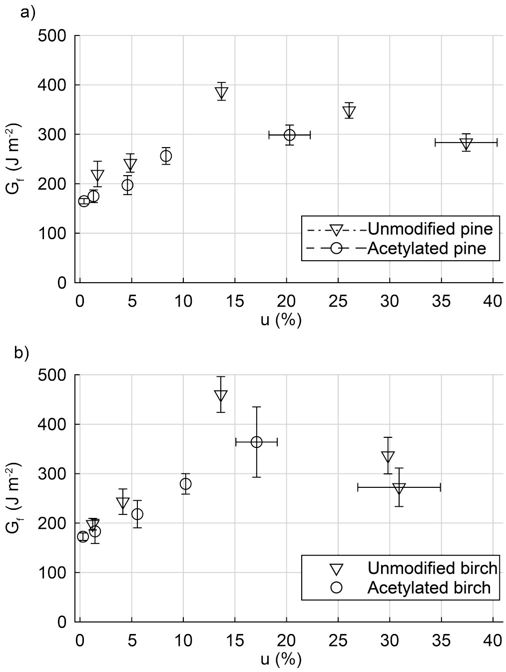

Figure 8: *The fracture energy (G_f) versus moisture content (u) for unmodified and acetylated Scots pine (a) and birch (b). Markers denote the mean values and error bars the standards deviations of the fracture energy. Moisture contents for acetylated wood are corrected according to Eq. 1, and for C5 the estimated intervals of the cell wall moisture contents, u_{cw}, are presented.*

wood. Results corresponding to moisture contents below 15% are presented for each test group in Figure 9, along with regression lines and confidence intervals with a 95% level of significance. The results obtained were found to align with previous results for unmodified and acetylated Scots pine (Forsman et al. 2020) and birch (Lai and Plönning 2019). By means of a one-way analysis of covariance using the function aoctool in MATLAB (MATLAB R2017b, The MathWorks Inc., Natick, MA, US), the slopes of the regression lines for the unmodified and acetylated wood were compared, and were found to be different at a level of significance greater than 98% and 99% for Scots pine and birch, respectively. For dry wood, i.e. comparing the intercepts of the regression lines, no difference was found for birch while a significant difference at a level of 97% was suggested for Scots pine.

The findings demonstrated larger fracture energy for unmodified wood compared to acetylated wood for the moisture content levels investigated (0%–15%). Thus, the increased brittleness observed for acetylated wood when compared to unmodified wood at equal RH levels cannot solely be explained by the reduced hygroscopicity of acetylated wood, i.e. that the moisture content was lower. Previous research (Phan et al. 2017) has suggested that the crack-bridging mechanism is predominant for the increased fracture energy at higher moisture contents, while the effect of the micro-cracking phenomenon is suggested to remain constant. This statement is also supported by scanning electron microscope images of fracture surfaces presented by Reiterer and Tschegg (2002). In the present study, the discrepancy in

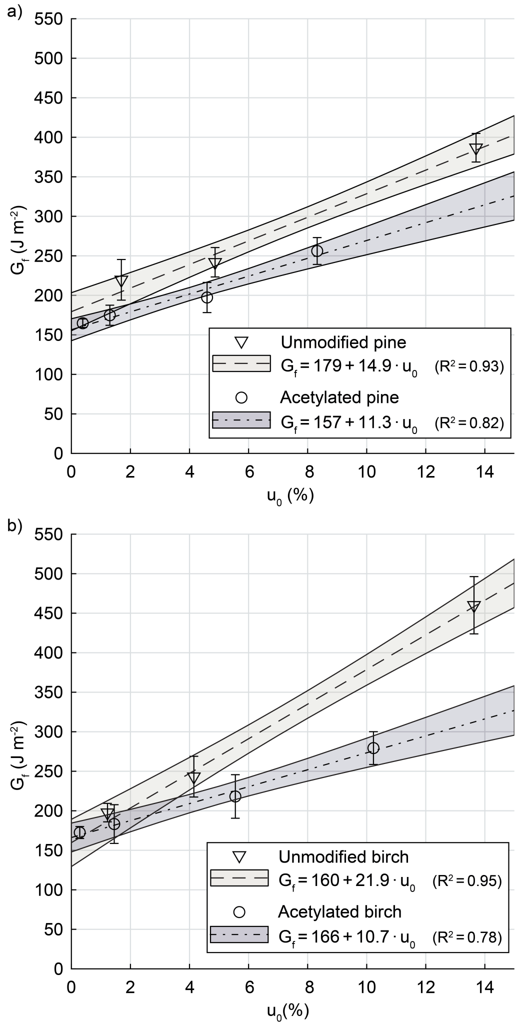

Figure 9: *Regression lines along with confidence intervals for the fracture energy (G_f) versus the moisture content (u_0) for Scots pine (a) and birch (b), fitted to moisture contents below 15%. Markers denote the mean values and error bars the standards deviation of the fracture energy.*

fracture energy between the unmodified and the acetylated wood was found to increase along with the moisture content. Considering the findings in Phan (2017) and Reiterer and Tschegg (2002), it could, thus, be hypothesized that this difference is due to differences in crack-bridging.

Fracture energy versus relative humidity

To provide an estimation of when the impact of an increased brittleness should be considered in structural design of acetylated wood, as well as the magnitude of that impact, the fracture energy for acetylated and unmodified wood were compared at equal RH levels. Accordingly, Figure 10 illustrates the difference in mean fracture energy for acetylated wood relative to unmodified wood at different RH levels, along with 95% confidence intervals. It is worth noting that the impact at different RH levels varied in a similar manner for Scots pine and birch. The following three observations were made for both wood species: (1) At RH levels up to 97%, a statistical significance of a reduced fracture energy for the acetylated wood was found. (2) The largest impact on the fracture energy was identified at 75% RH, where the loss in fracture energy for the acetylated wood compared to the unmodified wood was approximately 50%. (3) No significant impact on the fracture energy was found for the water-saturated samples.

As previously discussed, the lower fracture energy observed for acetylated wood compared to unmodified wood at equal RH levels can partly, but not solely, be explained by the reduced hygroscopicity of acetylated wood combined with the moisture-dependency of the fracture energy: Since the equilibrium moisture content of acetylated wood is lower compared to unmodified wood (according to the sorption isotherms), acetylated

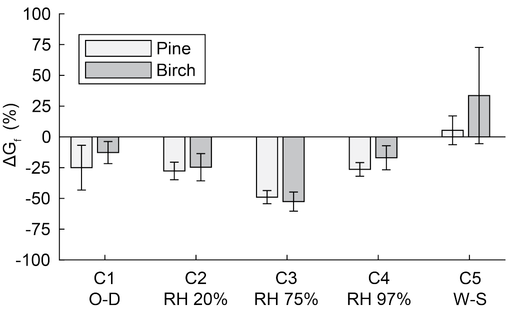

Figure 10: *The change in mean fracture energy (G_f) for acetylated wood relative to unmodified wood for examined conditions. The error bars represent a 95% confidence interval, based on a two-sample t-test assuming unequal variances.*

wood will demonstrate a lower fracture energy (according to the moisture-dependency of the fracture energy). This observation is important in structural design, where the influence of moisture on the material parameters of wood is typically considered by modification factors linked to serviceability classes. Such serviceability classes are typically defined in structural design codes and standards based on the RH levels of the ambient climate. The observations presented in this study suggested an increased brittleness for all outdoor conditions, and due to the yearly fluctuation of the RH, the worst impact should be considered, i.e. a reduced fracture energy of 50%. As an example, the design provisions of Eurocode 5 for dowel type joints include the use of minimum edge distances in order to avoid brittle failure modes. The validity of such provision for acetylated wood can be questioned, based on the findings of this research. On the other hand, acetylated wood exhibits an increased dimensional stability and less moisture induced stress gradients. This indicates lower stress concentrations for applications where acetylated wood is used, which is when the brittleness would be decisive. Moreover, the reduced hygroscopicity of acetylated wood will have further implications on structural design. For example, reduced creep and mechano-sorptive creep has been found for acetylated wood compared to unmodified wood (Epmeier and Kliger 2005), and a study on Accoya wood (Marcroft et al. 2014) has shown a decreased impact on strength values at conditions corresponding to service class 3.

Conclusions

A clear moisture-dependency of the fracture energy was demonstrated for unmodified as well as acetylated wood, and a lower fracture energy for acetylated wood when compared to unmodified wood was found also at equal moisture contents. Thus, previous findings, demonstrating an increased brittleness of acetylated wood when compared to unmodified wood at equal RH levels, cannot solely be a consequence of the reduced hygroscopicity of acetylated wood. Nevertheless, the reduced hygroscopicity of acetylated wood along with the moisture-dependency of the fracture energy, contributes to the lower fracture energy found for acetylated wood when compared to unmodified wood at equal RH levels; acetylated wood exhibits a lower moisture content, thus, a lower fracture energy. This observation is of importance in practical applications, such as structural design. In this study, acetylated wood demonstrated significantly lower values of the fracture energy at relative humidity levels up to 97%. The largest impact was identified as approximately 50%, observed at 75%

relative humidity. As the relative humidity outdoors has a yearly fluctuation, the worst impact should be regarded in the design of load-bearing structures.

Acknowledgements

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References

Beck, G., Thybring E.E., Thygesen L.G. and Hill, C. (2018). Characterization of moisture in acetylated and propionylated radiata pine using low-field nuclear magnetic resonance (LFNMR) relaxometry. Holzforschung 72:225–233.

Bongers, F. and Beckers, E. (2003). Mechanical properties of acetylated solid wood treated on pilot plant scale. In: Van Acker, J. and Hill, C. (Eds.), Proceedings of the First European Conference on Wood Modification. Ghent University, Ghent, pp. 241–350.

Brelid, P.L. (2013). Benchmarking and state of the art for modified wood. SP Technical Research Institute of Sweden, Stockholm.

Dreher, W.A., Goldstein, I.S. and Cramer, G.R. (1964). Mechanical properties of acetylated wood. For. Prod. J. 14:66–68.

Dourado, N., de Moura, M.F.S.F., Xavier, J. and Pereira, F.A.M. (2015). A New Procedure for Mode I Fracture Characterization of Cement-Based Materials. Strain 51: 483–491.

Engelund, E.T., Thygesen, L.G., Svensson, S. and Hill, C.A.S. (2013). A critical discussion of the physics of wood-water interactions. Wood Sci. Technol. 47:141–161.

Epmeier, H. and Kliger, R. (2005). Experimental study of material properties of modified Scots pine. Holz Roh- Werkst. 63:430–436.

Forsman, K., Serrano, E., Danielsson, H. and Engqvist, J. (2020). Fracture characteristics of acetylated young Scots pine. Eur. J. Wood Prod. 78:693–703.

Giotra, K. (2014). Process for wood acetylation and product thereof, European Patent No. 2818287A1.

Glass, S.V., Boardman, C.R., Thybring, E.E. and Zelinka, S.L. (2018). Quantifying and reducing errors in equilibrium moisture content measurements with dynamic vapor sorption (DVS) experiments. Wood Sci. Technol. 52:909–927.

Glass, S.V., Boardman, C.R. and Zelinka, S.L. (2017). Short hold times in dynamic vapor sorption measurements mischaracterize the equilibrium moisture content of wood. Wood Sci. Technol. 51:243–260.

Goldstein, I.S., Jeroski, E.B., Lund, A.E., Nielson, J.F. and Weaver, J.W. (1961). Acetylation of wood in lumber thickness. For. Prod. J. 11:363–370.

Greenspan, L. (1977). Humidity Fixed Points of Binary Saturated Aqueous Solutions. J. Res. Natl. Bur. Stand. 81:89–96.

Gustafsson, P.J. (2003). Fracture perpendicular to grain – Structural applications. In: Thelandersson, S. and Larsen H.J (Eds.), Timber engineering. John Wiley Sons, Chichester, pp. 114–115.

Hunt, C.G., Zelinka, S.L., Frihart, C.R., Lorenz, L., Yelle, D., Gleber, S.C., Vogt, S. and Jakes, J. E. (2018). Acetylation increases relative humidity threshold for ion transport in wood cell walls – A means to understanding decay resistance. Int. Biodeterior. Biodegrad. 133: 230–237.

Kollmann, F.F.P. (1968). Mechanics and Rheology of Wood. In: Kollmann, F.F.P. and Côté W.A. (Eds.), Principles of Wood Science and Technology. Springer-Verlag, Berlin, Heidelberg, pp. 292–419.

Lai, G. and Plönning, S. (2019). Fracture characteristics of acetylated birch – Experimental and numerical studies, Master’s thesis. Lund, Lund University.

Larsson, P. and Simonson, R. (1994). A study of strength, hardness and deformation of acetylated Scandinavian softwoods. Holz Roh- Werkst. 52:83–86.

Majano-Majano, A., Lara-Bocanegra, A.J., Xavier, J. and Morais, J. (2019). Measuring the

Cohesive Law in Mode I Loading of Eucalyptus globulus. Materials 12:23.

Majano-Majano, A., Hughes, M. and Fernandez-Cabo, J.L. (2012). The fracture toughness and properties of thermally modified beech and ash at different moisture contents. Wood Sci. Technol. 46:5–21.

Marcroft J., Bongers F., Perez F.P., Alexander J. and Harrison I. (2014). Structural Performance of Accoya® Wood under Service Class 3 Conditions. In: Aicher, S., Reinhardt, H.W. and Garrecht, H. (Eds.), Materials and Joints in Timber Structures, RILEM Bookseries, Vol. 9. Springer, Dordrecht, pp. 615–627.

Nordtest method (1993). Wood: Fracture energy in tension perpendicular to the grain (NT BUILD 422).

Ostapska, K. and Malo, K.A. (2020). Wedge splitting test of wood for fracture parameters estimation of Norway Spruce, Eng. Frac. Mech., DOI: 10.1016/j.engfracmech.2020.107024 (Epub ahead of print).

Plötze, M. and Niemz, P. (2011). Porosity and pore size distribution of different wood types as determined by mercury intrusion porosimetry. Eur. J. Wood Wood Prod. 69:649–657.

Rowell, R.M. (2006). Chemical modification of wood: A short review. Wood Mater. Sci. Eng. 1:29–33.

Rowell, R.M. (1996). Physical and mechanical properties of chemically modified wood. In: Hon, D.N.S. (Ed.), Chemical Modification of Lignocellulosic Materials. Routledge, New York, pp. 295–310.

Rowell, R.M. (1983). Chemical Modification of Wood. For. Prod. Abstr. 6:363–382.

Rowell, R.M. and Dickerson, J.P. (2014). Acetylation of wood. In: Schultz, T.P., Goodell, B. and Nicholas, D.D (Eds.), Deterioration and Protection of Sustainable Biomaterials. American Chemical Society, Washington DC, pp. 301–327.

Reiterer, A. and Sinn, G. (2002). Fracture behaviour of modified spruce wood: A study using linear and non linear fracture mechanics. Holzforschung 56:191–198.

Reiterer, A. and Tschegg, S. (2002). The influence of moisture content on the mode I fracture behaviour of sprucewood. J. Mater. Sci. 37:4487–4491.

Schwanninger, M., Stefke, B. and Hinterstoisser, B. (2011). Qualitative assessment of acetylated wood with infrared spectroscopic methods. J. Near Infrared Spectrosc. 19:349–357.

Serrano, E. and Enquist, B. (2005). Contact-free measurement and non-linear finite element analyses of strain distribution along wood adhesive bonds. Holzforschung, 59(6):641–646. Smith, I. and Chui, Y.H. (1994). Factors affecting mode I fracture energy of plantation-grown red pine. Wood Sci. Technol. 28:147–157.

Telkki, V.V., Yliniemi, M. and Jokisaari, J. (2013). Moisture in softwoods: fiber saturation point, hydroxyl site content, and the amount of micropores as determined from NMR relaxation time distributions. Holzforschung 67:291–300.

Thybring, E.E. (2013). The decay resistance of modified wood influenced by moisture exclusion and swelling reduction. Int. Biodeterior. Biodegrad. 82:87–95.

Williams, D.R. (1995). The characterisation of powders by gravimetric water vapour sorption. Int. Labmate 20:40–42.

Zelinka, S.L., Ringman, R., Pilgård, A., Thybring, E.E., Jakes, J.E. and Richter, K. (2016). The role of chemical transport in the brown-rot decay resistance of modified wood. Int. Wood Prod. J. 7:66–70.

Paper C

BRITTLENESS OF DOWEL-TYPE CONNECTIONS MADE FROM ACETYLATED WOOD

Karin Forsman[1], Erik Serrano[2], Henrik Danielsson[3]

ABSTRACT: This paper presents an investigation of the effects of acetylation on the brittleness of dowel-type connections. The aim is to investigate if the current design provisions of Eurocode 5 can be safely applied to acetylated Scots pine, or should be revised to account for the increased brittleness of such wood. Dowel-type connections loaded perpendicular and parallel to the grain were examined experimentally. To highlight the effect of the increased brittleness, end- and edge-distances were varied and the results compared with theoretical results based on the design criteria of Eurocode 5. In line with the increased density of acetylated wood, the results showed an increased embedment strength for acetylated wood compared to unmodified wood. For loading parallel to the grain, acetylated wood failed in a brittle manner regardless of the end-distance. To account for the increased embedment strength parallel to the grain, reinforcements are recommended. For loading perpendicular to the grain, both unmodified and acetylated wood failed due to splitting, indicating an insufficiency of current design provisions for loading at an angle to the grain. In agreement with previous studies, indicating a lower fracture energy for acetylated wood, a decreased splitting capacity was observed when compared to unmodified wood.

KEYWORDS: Acetylation, Dowel-type connections, Wood fracture, Splitting, Scots pine, Brittle failure

1 BACKGROUND

Chemical modification methods are known to increase both durability and dimensional stability of wood. However, since the chemical constitution of the cell wall polymers is changed, some mechanical properties are also affected [1]. Previous studies have demonstrated that acetylated wood becomes significantly more brittle [2–4]. In structural applications, an increased brittleness is important to consider due to the occurrence of stress concentrations, *e.g.* in mechanical joints [5].

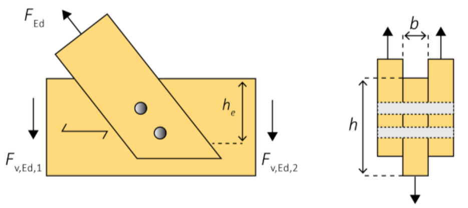

Figure 1: *Dowel-type connection loaded perpendicular to the grain.*

Tensile stresses perpendicular to the grain should be avoided in the design of timber structures, but this is not always possible. Figure 1 illustrates a dowel-type connection loaded *perpendicular to the grain*, which can fail by splitting due to crack initiation and propagation along the grain [6–7]. Based on Linear Elastic Fracture Mechanics (LEFM), a design criterion for the verification of joints loaded perpendicular to the grain is implemented in Eurocode 5 (EC5), [8], stating that the maximum shear force at the connection, $F_{v,Ed}$, should satisfy:

$$F_{v,Ed} \leq F_{90,Rd} \quad (1)$$

with $F_{v,Ed}=\max(F_{v,Ed,1}, F_{v,Ed,2})$, defined according to Figure 1. The characteristic shear force capacity, $F_{90,Rk}$, for a connection with a metal dowel-type fastener is determined by [8]:

$$F_{90,Rk}=14b\sqrt{\frac{h_e}{1-h_e/h}} \quad (2)$$

According to this design criterion, only geometrical parameters impact the shear force capacity of the joint.

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However, the original form of Equation (2) has been presented as [7]:

$$F_{90,\mathrm{Rk}}=b\sqrt{\frac{GG_c}{0.6}}\sqrt{\frac{h_e}{1-h_e/h}} \quad (3)$$

where G_c is the critical energy release rate and G the longitudinal shear modulus. Thus, in the simplified criterion, Equation (2), assumptions regarding linear-elastic fracture properties are already imposed, based on results from tests conducted on softwoods. Hence, the generalization of this criterion is restricted.

When designing dowel-type connections loaded *parallel to the grain*, brittle failure modes illustrated in Figure 2 must be regarded [6]. In EC5, [8], the design of dowel type joints is based on the Johansen yield theory, which assumes ductile failure modes by plastic deformations of the dowel and/or the timber. Thus, to take into account the brittle failure modes, minimum edge- and end-distances are prescribed. Again, these design criteria are based on implicitly assumed material characteristics and should therefore be used cautiously.

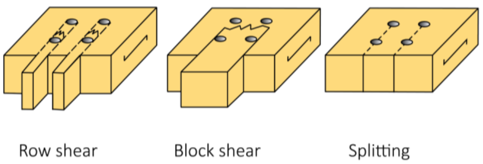

Figure 2: *Brittle failure modes for dowel-type connections loaded parallel to the grain.*

From the above examples, it is obvious that the design criteria of EC5 to prevent premature brittle failure in dowel-type connections, are implicitly based on assumptions regarding the fracture characteristics of the material. Since acetylated wood exhibits an increased brittleness, current design provisions might not account for this. This research aims at investigating if design provisions should be revised for acetylated wood.

2 METHOD

2.1 MATERIAL

In order to study how acetylation affects the brittleness of dowel-type connections, timber members of both unmodified and acetylated Scots pine (*Pinus sylvestris*) were examined. The examined material originated from the sawmill Isojoen Saha, located in Finland. The modified boards were acetylated in a proprietary industrial-scale process, at Accys Technology in Arnhem, the Netherlands, using the standard process in the commercial production process of Accoya radiata pine [9]. No adjustments were made to the acetylation process, neither regarding time, temperature nor concentrations. The modified boards were analysed using near infrared spectroscopy [10] and the acetyl content was found to be approximately 20%. Specimens were stored in a climate chamber with a relative humidity of 60% and a temperature of 20°C before testing, until reaching moisture equilibrium. The mean densities of the unmodified and the acetylated boards were 484 kg/m^3 and 493 kg/m^3, respectively. For unmodified wood, the moisture content determined by the oven-dry method was 10%. The moisture content of the acetylated wood was approximately 3.4 %, determined in the same manner but adjusted to take into account the increase in dry mass as a consequence of the acetylation [11].

2.2 DOWEL-TYPE CONNECTIONS

Dowel-type connections loaded perpendicular and parallel to the grain were tested. In order to promote brittle failure modes and avoid plasticity in the fasteners, the timber members were assigned a sufficiently small thickness in relation to the dowel diameter. No deformation of the dowel was identified during or after testing. A material testing system (MTS322 Test Frame) was used, and the load was recorded by a load cell (MTS 500 kN) with a resolution of 0.005 kN. Displacements were recorded by LVDT sensors (RDP ± 10 mm) with a resolution of 0.003 mm. A dowel with a diameter d = 12 mm was used in all tests.

2.2.1 Parallel to the grain loading

To prevent brittle failures for dowel-type connections loaded parallel to the grain, a minimum distance from the dowel to the loaded end, $a_{3,t}$, is prescribed by EC5 [8]:

$$a_{3,t} = \max[7d;\ 80\ \mathrm{mm}] \quad (4)$$

where d is the diameter of the dowel. In this study, dowel-type connections loaded parallel to the grain were examined as illustrated in Figure 3. With the considered dowel dimension, a minimum end-distance $a_{3,t} = 7d$ is thus prescribed in EC5.

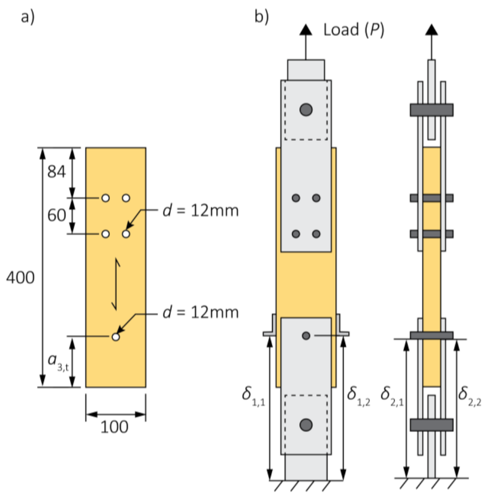

Figure 3 *Geometry of specimens (a) and test set-up (b) for dowel connections loaded parallel to the grain, where the load (P) was applied with an end-distance $a_{3,t}$ to the loaded edge.*

Table 1: *The number of specimens tested in loading parallel to the grain for each end-distance $a_{3,t}$. According to EC5, a minimum end-distance of $a_{3,t}$ = 7d is recommended (d=12 mm).*

$a_{3,t}$ (mm)	$a_{3,t}/d$	Unmodified	Acetylated
60	5	4	-
84	7	4	4
108	9	4	4
132	11	-	4

Three end distances were studied for each test group, cf. Table 1. The load, P, was applied at a rate of 1 mm/min by a displacement-controlled movement of the crosshead of the testing machine. Deformations were measured for the wood, in line with the dowel, and considered by the average of $\delta_{1,1}$ and $\delta_{1,2}$. To ensure that no deformation of the dowel occurred, displacements $\delta_{2,1}$ and $\delta_{2,2}$ were also monitored.

2.2.2 Perpendicular to the grain loading

For loading perpendicular to the grain, EC5 prescribes a minimum edge-distance $a_{4,t}$ [8]:

$$a_{4,t} = \max[(2+2\sin\alpha)d;\ 3d] \qquad (5)$$

where d is the diameter of the dowel and α the angle between the loading direction and the grain. In this study, specimens were loaded perpendicular to the grain according to Figure 4. With the considered loading direction and dowel dimension, a minimum edge-distance $a_{4,t}$ = 4d is suggested in EC5. In this study two edge-distances were considered, and the number of specimens within each test group is presented in Table 2. The load, P, was applied by a displacement-controlled movement of the crosshead of the testing machine at a rate of 1 mm/min, and the specimens were loaded until failure. Locations of extensometers used are shown in Figure 4. The displacement of the dowel was considered by an averaged value between $\delta_{1,1}$ and $\delta_{1,2}$.

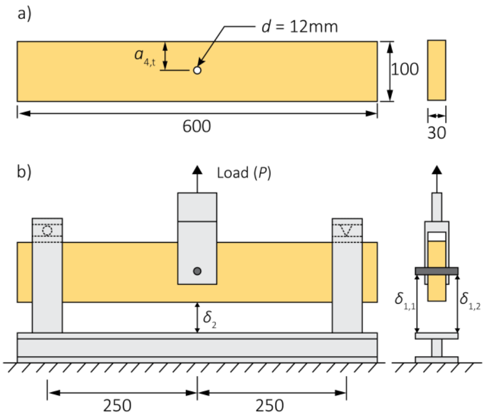

Figure 4: *Geometry of specimens (a) and test set-up (b) for dowel connections loaded perpendicular to the grain, with the load (P) applied at an edge-distance $a_{4,t}$.*

Table 2: *The number of specimens tested in loading perpendicular to the grain for each edge-distance $a_{4,t}$. According to EC5, a minimum edge-distance of $a_{4,t}$ = 4d is recommended (d = 12 mm).*

$a_{4,t}$ (mm)	$a_{4,t}/d$	Unmodified	Acetylated
48	4	4	4
64	5.33	4	4

2.2.3 Evaluation of ductility

To evaluate the brittleness of the examined connections, a measurement of the ductility (displacement-capacity of the post-linear elastic response) was applied:

$$D_f = \frac{u_f}{u_y} \qquad (6)$$

where u_f is the displacement at failure, and u_y the displacement at the yielding point. In this study, failure of a specimen was defined by a 20% load decrease from the ultimate load P_u. The yielding point was defined according to Figure 5, i.e. by the intersection of the load-displacement response and the linear elastic response offset 0.1 mm. The linear elastic response was in turn defined by the slope k_e, evaluated for load values between 40% – 60% of the ultimate load.

In the literature, various definitions for the yielding point and the failure point have been suggested, e.g. by defining the failure displacement as the displacement at maximum load. The approach used here was chosen to quantify the displacement capacity also for cases involving a slightly diminishing load bearing capacity after maximum load. To categorise the ductility of the load-displacement responses, classifications based on D_f have been reported according to Table 3 [12].

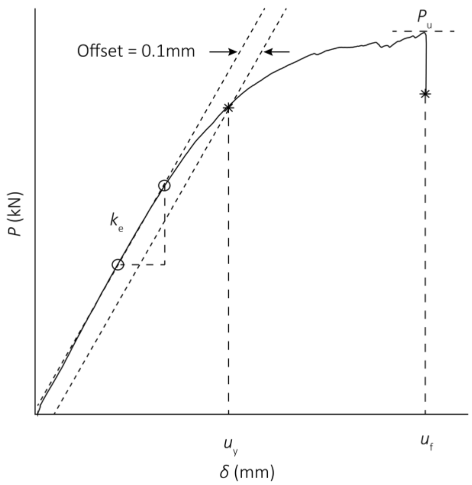

Figure 5: *Definition of yielding point (u_y) and failure point (u_f) for a typical load (P) versus displacement (δ) response, where P_u is the ultimate load and k_e is the linear elastic slope.*

__Table 3:__ Classifications [12] of ductility based on the ductility ratio D_f, defined in Equation (6).

Classification	D_f
Brittle	$D_f \leq 2$
Low ductility	$2 < D_f \leq 4$
Moderate ductility	$4 < D_f \leq 6$
High ductility	$D_f > 6$

2.2.4 Embedment strength

To facilitate the analysis of the examined dowel-type connections, the embedment strength parallel and perpendicular to the grain was determined for the unmodified and the acetylated Scots pine. For each direction and material, four samples were examined. Specimens were tested according to Figure 6, i.e. in accordance with ISO/FDIS 10984-2 [13]. The load was applied at a rate of 1 mm/min by a displacement-controlled movement of the crosshead of the testing machine. The displacement of the dowel was considered by an averaged value between $\delta_{1,1}$ and $\delta_{1,2}$. Specimens were loaded until failure, or until the displacement reached 5 mm. Based on the ultimate load P_u, (or the maximum load reached within 5 mm displacement) the embedment strength $f_{h,\alpha}$ was evaluated by:

$$f_{h,\alpha} = \frac{P_u}{t\,d} \qquad (8)$$

where t is the thickness of the specimen, and d the diameter of the dowel.

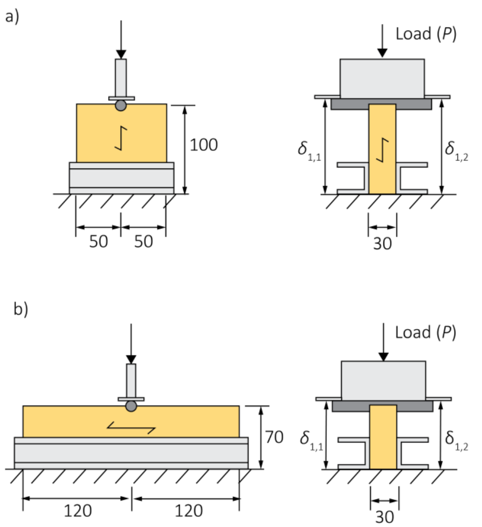

Figure 6: *Test set-up for determination of embedment strength, parallel (a) and perpendicular (b) to the grain, according to ISO/FDIS 10984-2 [13] with a dowel with a diameter of 12 mm.*

2.3 COMPARISON WITH EUROCODE 5

To assess whether current EC5 design provisions are appropriate for acetylated wood, results for the examined dowel-type connections were evaluated against EC5 predictions. In EC5, the characteristic embedment strength, $f_{h,\alpha,k}$, at an angle, α, to the grain is given by [8]:

$$f_{h,\alpha,k} = \frac{f_{h,0,k}}{k_{90}\sin^2\alpha + \cos^2\alpha} \quad [\text{N/mm}^2] \qquad (9a)$$

$$f_{h,0,k} = 0.082(1 - 0.01d)\rho_k \quad [\text{N/mm}^2] \qquad (9b)$$

$$k_{90} = 1.35 + 0.015d \quad \text{for softwood} \qquad (9c)$$

where ρ_k is the characteristic density and with d given in mm. In this study, experimental results were compared with calculated values based on the EC5-formulae but using the mean densities of the unmodified and acetylated Scots pine (after conditioning the specimens at a relative humidity of 60% and a temperature of 20 ºC).

Based on the embedment strength, the load-bearing capacity of a dowel-type connection can be estimated. As connections studied herein were designed to avoid plasticity in the fasteners, the governing failure mode should be embedment failure according to Johansen's yield theory, or brittle failure modes. According to EC5 the characteristic load-bearing capacity (per shear plane) associated with embedment failure, $F_{v,Rk}$, is given by [8]:

$$F_{v,Rk} = f_{h,\alpha,k}\, t\, d \qquad (10)$$

where $f_{h,\alpha,k}$ is the characteristic embedment strength for loading at an angle α to the grain, t the thickness of the timber member, and d the diameter of the dowel.

For dowel-type connections loaded parallel to the grain, the only brittle failure mode covered in EC5 is block-shear (cf. Figure 2). As described in Annex A of EC5 [8], the load-bearing capacity is restricted due to block shear by:

$$F_{bs,Rk} = \max \begin{cases} 1.5\, A_{net,t} f_{t,0,k} \\ 0.7\, A_{net,v} f_{v,k} \end{cases} \qquad (11)$$

where $f_{t,0,k}$ is the characteristic tensile strength parallel to the grain, and $f_{v,k}$ the characteristic shear strength. Both these nominal strengths are most likely affected by the brittleness of the material. For dowel-type connections, $A_{net,t}$ and $A_{net,v}$ are the effective areas of the head tensile plane and the lateral shear planes, respectively [8]. For loading perpendicular to the grain, splitting is considered in EC5 by Equation (2).

3 RESULTS AND DISCUSSION

3.1 EMBEDMENT STRENGTH

The embedment strength is primarily affected by timber density and dowel dimension. Since acetylation of wood is associated with an increased density, an increased embedment strength for acetylated wood could be suspected. Results from this study are presented in Table 4, presenting mean values of the embedment strength parallel and perpendicular to the grain ($f_{h,\alpha,m}$), along with values based on Equations (9a-c) ($f_{h,\alpha,EC5}$). As expected, comparing acetylated and unmodified wood in loading parallel to the grain, a significantly increased embedment strength was found for the acetylated wood. For loading perpendicular to the grain, no significant difference was found, thus, the embedment strength perpendicular to the grain can be considered unaltered.

Comparing the test results against the EC5-predictions, a parameter β was introduced, representing the embedment strength based on the EC5-approach, divided by the experimentally found mean embedment strength. For perpendicular to grain loading, estimates based on mean density values provided embedment strengths 5% – 11% lower than the mean embedment strength from the tests. However, for loading parallel to the grain of acetylated wood, the EC5-approach severely underestimated (−24%) the embedment strength, indicating an increased strength not only due to the increased density. Presented β-values indicate that the EC5-approach seems to be on the safe side, and thus, in this case it may be used also for acetylated Scots pine. It should, however, be noted that previous studies have shown a strong variability dependent on what method is used to determine the embedment strength [14], and variability is, of course an important factor in estimating characteristic values.

Table 4: *Test results and EC5 estimates of the embedment strength $f_{h,\alpha}$ in (MPa), and the ratio $\beta = f_{h,\alpha,EC5}/f_{h,\alpha,m}$. The difference in embedment strength between unmodified and acetylated wood, $\Delta f_{h,90}$, is marked (*) if significant (p<0.05).*

		$f_{h,\alpha,m}$	$f_{h,\alpha,EC5}$	β
Parallel	Unmodified	**36**	35	0.97
(α = 0º)	Acetylated	**47**	36	0.76
	$\Delta f_{h,0}$	+ 31%*		
Perpendicular	Unmodified	**24**	23	0.95
(α = 90º)	Acetylated	**26**	23	0.89
	$\Delta f_{h,90}$	(+ 8%)		

3.2 DOWEL CONNECTION LOADED PARALLEL TO THE GRAIN

Load-displacement responses for dowel-type connections loaded parallel to the grain are shown in Figure 7, and the evaluated ductility D_f for each test group is presented in Table 5. For unmodified wood (Figure 7a) with the recommended minimum end-distance $a_{3,t}$ = 7d, the response was classified as moderately ductile (D_f = 5), attributed to embedment failure followed by splitting (Figure 8a). An increased end-distance, $a_{3,t}$ = 9d, did not have a considerable effect on the ductility, while a decreased end-distance, $a_{3,t}$ = 5d, resulted in responses classified by low ductility. For acetylated wood (Figure 7b), a design using the recommended end-distance resulted in brittle failures (D_f = 1.1). An increased end-distance increased the ductility, but failures were still considered brittle (D_f < 2.0). The primary failure mode for acetylated wood was block/row shear, as shown in Figure 8b. This feature has also been indicated in a previous study of acetylated wood [15], where brittle failure modes attributed to block/row shear dominated.

Table 5: *Ductility, D_f, evaluated for dowel-type connections loaded parallel to the grain.*

$a_{3,t}$	5d	7d	9d	11d
Unmodified	2.2	5.0	4.9	-
Acetylated	-	1.1	1.7	1.7

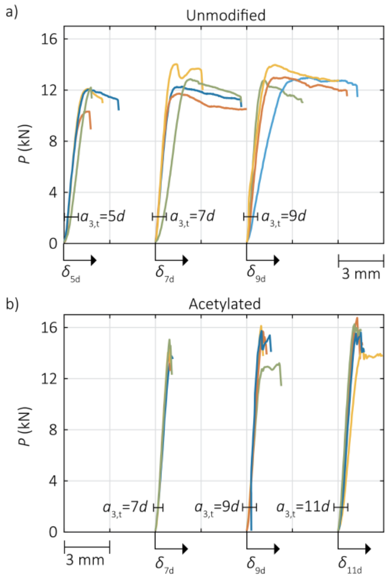

Figure 7: *Load-displacement responses for dowel-type connections loaded parallel to the grain, for unmodified (a) and acetylated wood (b) with varying end-distances $a_{3,t}$ (d=12 mm).*

a) b)

Figure 8: *Typical failure modes observed: for unmodified wood, splitting occurred (a), while block/row shear was observed for acetylated wood (b).*

Table 6: *Mean load-bearing capacity for connections loaded parallel to the grain, $F_{0,m}$ in (kN). The ratio β represents the EC5 estimation (Equation (10) based on either the mean or the EC5 estimate of the embedment strength) divided by the mean load-bearing capacity. The difference in load-bearing capacity between unmodified and acetylated wood $\Delta F_{0,m}$ is marked (*) if significant ($p<0.05$).*

$a_{3,t}$		5*d*	7*d*	9*d*	11*d*
Unmodified	$F_{0,m}$	**11.7**	**12.7**	**13.2**	-
	β_m	1.1	1.0	0.98	-
	β_{EC5}	1.1	0.99	0.95	-
Acetylated	$F_{0,m}$	-	**14.4**	**15.2**	**15.7**
	β_m	-	1.2	1.1	1.1
	β_{EC5}	-	0.89	0.84	0.82
$\Delta F_{0,m}$			+13%*	+15%*	

Mean values of the load-bearing capacity for connections loaded parallel to the grain, $F_{0,m}$, are presented in Table 6. Comparing the load-bearing capacity of connections made from unmodified and acetylated wood, the acetylated wood demonstrated a significantly increased load-bearing capacity. This was an expected result, in line with the previous observation of an increased embedment strength parallel to the grain.

To compare the results against estimations based on the EC5-approach, again a ratio β was introduced, representing the EC5 estimation, i.e. Equation (10) based on either the mean embedment strength $f_{h,0,m}$ or the estimated embedment strength $f_{h,0,EC5}$ (Table 4), divided by the experimentally found mean load-bearing capacity. For the ratio based on the estimated embedment strength, β_{EC5}, conservative values of the load-bearing capacity were achieved ($\beta_{EC5} \leq 1$). However, the load-bearing capacity based on the observed embedment strength, β_m, was non-conservative ($\beta_m > 1$) for the acetylated wood. This is most likely attributed to the premature brittle failures observed; due to the increased embedment strength of acetylated wood, the load-bearing capacity will be limited by brittle failure modes, cf. Equation (11). To utilize the increased embedment strength of dowel-type connections made from acetylated wood, but still ensure ductile failures, reinforced joints are recommended. Alternatively, yielding of dowels prior to brittle failure modes can be promoted by using dowels with a lower steel grade.

3.3 DOWEL CONNECTION LOADED PERPENDICULAR TO THE GRAIN

Load-displacement responses for loading perpendicular to the grain are shown in Figure 9, and the evaluated ductility (D_f) is presented in Table 7. For unmodified wood, both edge-distances yielded responses with low ductility (D_f = 3), and the failure modes were attributed to splitting along the grain (Figure 10a). For acetylated wood, the recommended edge-distance according to EC5 resulted in clearly brittle responses (D_f = 1.3), caused by splitting (Figure 10b). An increased edge-distance increased the ductility ratio (D_f = 2) but the failure modes were still considered brittle.

Table 7: *Ductility, D_f, evaluated for dowel-type connections loaded perpendicular to the grain.*

$a_{4,t}$	4*d*	5.33*d*
Unmodified	3.1	3.0
Acetylated	1.3	2.0

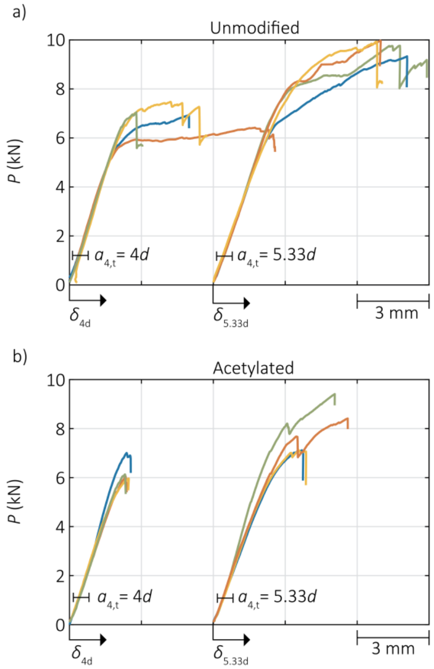

Figure 9: *Load-displacement responses observed for connections loaded perpendicular to the grain for unmodified (a) and acetylated wood (b) with varying edge-distances $a_{4,t}$ (d=12 mm).*

Figure 10: *Typical failure modes observed for unmodified (a) and acetylated wood (b).*

__Table 8:__ Mean load-bearing capacity for connections loaded perpendicular to the grain, $F_{90,m}$ in (kN). The ratio β represents the EC5 estimation (Equation (10) based on either the mean or the EC5 estimate of the embedment strength) divided by the mean load-bearing capacity. The difference in load-bearing capacity between unmodified and acetylated wood Δ $F_{90,m}$ is marked (*) if significant (p<0.05).

$a_{4,t}$		4*d*	5.33*d*
Unmodified	$F_{90,m}$	**6.97**	**9.75**
	β_m	1.2	0.89
	β_{EC5}	1.2	0.84
Acetylated	$F_{90,m}$	**6.29**	**8.01**
	β_m	1.5	1.2
	β_{EC5}	1.3	1.1
Δ $F_{90,m}$		(-10%)	-18%*

Mean values of the load-bearing capacity for dowel-type connections loaded perpendicular to the grain, $F_{90,m}$, are presented in Table 8. Comparing unmodified and acetylated wood, no significant difference in load-bearing capacity was identified for the recommended edge-distance $a_{4,t}$ = 4*d*. However, for an increased edge-distance, acetylated wood demonstrated a significantly lower capacity. To evaluate the load-bearing capacity against the EC5-approach, a ratio β was again introduced, indicating the relation between the estimated values according to the EC5-approach, i.e. Equation (10) using either the observed embedment strength $f_{h,90,m}$ or the estimated embedment $f_{h,90,EC5}$ (Table 4), and the experimentally found mean load-bearing capacity. It was found that the capacity was over-estimated in all cases except for one: connections made from unmodified wood with an increased edge-distance.

In this study, the load-bearing capacity perpendicular to the grain was clearly limited by brittle failure modes rather than embedment strength. As presented in Equations (2)–(3), dowel-joints loaded perpendicular to the grain are vulnerable to splitting along the grain, a failure mode which to a large degree is dependent on the fracture characteristics of the material. Since previous studies [2–3] have demonstrated a decreased fracture energy for acetylated wood, an increased brittleness of mechanical joints made from modified wood can be expected. Regarding the effect of acetylation on the shear force capacity, a simple estimate can be made. Considering Equation (3), assuming that all other characteristics remain unaltered by the modification, a reduction of the fracture energy by 50% [2–3] would equal a reduced load-bearing capacity of approximately 30%. In Figure 11, the mean load-bearing capacity for unmodified and acetylated wood is presented along with estimates based on Equation (10) for embedment failure, and Equation (2) for splitting, including a reduced splitting capacity of 30%. As can be observed for the recommended edge-distance ($a_{4,t}$ = 4*d*), the splitting capacity according to EC5 is overestimated for both unmodified and acetylated wood. Further, the results indicate that a decreased splitting capacity should be considered for acetylated wood compared to unmodified wood.

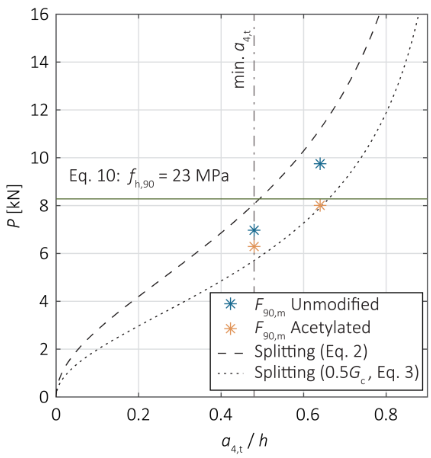

***Figure 11:** Mean load-bearing capacity of dowel-type connections loaded perpendicular to the grain, along with EC5 estimates based on embedment strength, and splitting capacity.*

4 CONCLUDING REMARKS

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

- Compared to unmodified wood, acetylated Scots pine demonstrated a significantly increased *embedment strength* parallel to the grain, while no difference could be found for perpendicular to the grain loading.
- For acetylated wood, using the measured density to estimate the embedment strength according to the EC5-approach provided reasonably conservative estimations of the embedment strength perpendicular to the grain (−11%), while the strength parallel to the grain was severely underestimated (−24%).
- For dowel-type connections loaded *parallel to the grain*, acetylated wood demonstrated an increased load-bearing capacity compared to unmodified wood.
- Using the EC5 design approach, a conservative estimation of the parallel to grain load-bearing capacity was obtained. However, connections made from acetylated wood experienced premature brittle failure modes. To achieve ductile responses, yet utilizing the increased embedment strength, reinforced joints are recommended. Alternatively, a lower steel grade could be used, to achieve yielding of dowels prior to block/row shear.

- The load-bearing capacity of dowel-type connections loaded *perpendicular to the grain* was lower for acetylated wood compared to unmodified wood. For both unmodified and acetylated wood, connections failed due to splitting along the grain. Results indicate an insufficiency of current design provisions for loading perpendicular to the grain, especially for acetylated wood where the impact of a decreased fracture energy should be regarded, and an increased edge-distance or reinforcement is recommended.

5 FURTHER RESEARCH

To develop further understanding of the brittleness of dowel-type connections made from acetylated wood, studies should be extended to include connections with multiple fasteners. The addition of dowels will increase stress concentrations, thus, recommended minimum distances between fasteners should be evaluated for acetylated wood. It is also important to remember that other design parameters may be affected by the acetylation process. As an example, studies on Accoya wood [16] have shown a decreased impact on strength values at conditions corresponding to service class 3, thus, indicating that k_{mod}-values for acetylated wood should be given consideration.

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REFERENCES

[1] R. M. Rowell. Chemical modification of wood: A short review. *Wood Material Science and Engineering,* 1:29-33, 2006.

[2] Forsman K., Serrano E., Danielsson H., Engqvist J.: Fracture characteristics of acetylated young Scots pine. *European Journal of Wood and Wood Products,* 78(4):693-703, 2020.

[3] Lai G., Plönning S.: Fracture characteristics of acetylated birch – Experimental and numerical studies (Master's thesis). TVSM-5000, 2019.

[4] Reiterer A., Sinn G.: Fracture behaviour of modified spruce wood: a study using linear and non linear fracture mechanics. *Holzforschung,* 56(2):191-198, 2002.

[5] P. J. Gustafsson. Fracture perpendicular to grain – Structural applications. In S. Thelandersson, H. J. Larsen, editors, *Timber engineering,* pages 103-130. John Wiley & Sons, Chichester, England, 2003.

[6] J. C. M. Schoenmakers. Fracture and failure mechanisms in timber loaded perpendicular to the grain by mechanical connections (Doctoral dissertation). ISBN: 978-90-386-2223-1, 2010.

[7] Ehlbeck J., Görlacher R.: E11 Joints loaded perpendicular to the grain. In: H. J. Blass, C. Sandhaas, editors, *Timber Engineering – Principles for Design,* pages 441-457. KIT Scientific Publishing, Karlsruhe, Germany, 2017.

[8] EN 1995-1-1. Eurocode 5, Design of timber structures: Part 1-1, General–Common rules and rules for buildings. 2004.

[9] K. Giotra: Process for wood acetylation and product thereof. European Patent No. 2818287A1. European Patent Office. 2014.

[10] Schwanninger M., Stefke B., Hinterstoisser B.: Qualitative assessment of acetylated wood with infrared spectroscopic methods. *J. Near Infrared Spectrosc.,* 19:349-357, 2011.

[11] E. E. Thybring: The decay resistance of modified wood influenced by moisture exclusion and swelling reduction. *Int. Biodeterior. Biodegrad*., 82:87-95, 2013.

[12] Smith I., Asiz A., Snow M., Chui Y. H.: Possible Canadian/ISO approach to deriving design values from test data. In *CIB-W18 Meeting in Florence,* 2006.

[13] ISO/DIS 10984-2. International Organization for Standardization, Timber Structures - Dowel-type Fasteners – Part 2: Determination of Embedding Strength and Foundation Values. 2009.

[14] Franke S., Magnière N.: Discussion of testing and evaluation methods for the embedment behaviour of connections. In *Proceedings of the International Network on Timber Engineering Research. INTER/47-7-1.* Bath, United Kingdom, 2014.

[15] S. Anderberg. Mechanical properties of chemical modified wood of load-bearing constructions (Master's thesis). TVBK-5257, 2016.

[16] Marcroft J., Bongers F., Perez F.P., Alexander J., Harrison I.: Structural Performance of Accoya® Wood under Service Class 3 Conditions. In Aicher S., Reinhardt HW., Garrecht H., editors, *Materials and Joints in Timber Structures, RILEM Bookseries, vol 9.* Springer, Dordrecht, Germany, 2014.