

Sini Metsä-Kortelainen

Differences between sapwood and heartwood of thermally modified Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*) under water and decay exposure



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# Differences between sapwood and heartwood of thermally modified Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*) under water and decay exposure

#### Sini Metsä-Kortelainen

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Sini Metsä-Kortelainen. Differences between sapwood and heartwood of thermally modified Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*) under water and decay exposure [Lämpökäsitellyn kuusen (*Picea abies*) ja männyn (*Pinus sylvestris*) pinta- ja sydänpuun käyttäytyminen vesi- ja lahorasituksessa]. Espoo 2011. VTT Publications 771. 58 p. + app. 64 p.

Keywords

decay, contact angle, heartwood, Norway spruce, sapwood, Scots pine, thermal modification, water absorption

#### **Abstract**

Thermal modification methods have been developed to increase the biological durability and dimensional stability of wood. The aim of this research was to study the differences between sapwood and heartwood of thermally modified Scots pine (*Pinus sylvestis*) and Norway spruce (*Picea abies*) under water and decay exposure. The effects of the modification temperature and wood coating were also examined.

Several tests were carried out in the laboratory and field with three different complementary research materials. The main research material consisted of sapwood and heartwood of Scots pine and Norway spruce thermally modified at temperatures of 170°C, 190°C, 210°C and 230°C. The reference materials were untreated sapwood and heartwood of pine and spruce, larch, bangkirai, Western red cedar, merbau and pressure-treated wood materials, depending on the test.

Thermal modification decreased the water absorption of sapwood and heartwood of spruce in relation to the modification temperature in a floating test. The water absorption of sapwood and heartwood of pine either decreased or increased, however, depending on the modification temperature. Pine sapwood absorbed more water, and very quickly, than the other wood materials, whilst pine heartwood was the most water-repellent material in the test. In general, the wettability of the thermally modified wood materials measured as contact angles only decreased with samples that had been modified at a very high modification temperature (230°C) compared with the untreated reference wood materials.

The decay resistance of thermally modified wood materials was studied in a laboratory brown-rot test with two fungi (*Coniophora puteana* and *Poria placenta*) and two incubation times (6 and 10 weeks), and in a soft-rot test with unsterile soil for 32 weeks. The fungal durability was also evaluated after 1, 2 and 9 years of exposure in the lap-joint field test. In general, the thermal modifi-

cation increased the fungal durability in all the cases: the higher the modification temperature, the higher the resistance to fungal attack. Significant differences were detected between the different tests and wood materials. A very high thermal modification temperature (230°C) was needed to achieve resistance against decay comparable to that of the durability classes 'durable' or 'very durable' in the soft-rot test. The brown-rot test resulted in slightly better durability classes than the soft-rot test, which means that, already at lower temperatures (190–210°C), thermal modification clearly increases resistance to brown-rot attack, especially with pine materials. The results after nine years of exposure in the lap-joint field test had a good correlation with the results in the laboratory test with brown-rot fungi.

The effects of the level of thermal modification and decay exposure on the bending strength of wood materials were investigated using small samples. On average, the thermal modification and fungal exposure both reduced the strength. The effect of decay exposure on strength was more significant however. It can be concluded that untreated wood material is stronger than thermally modified wood material until the wood is exposed to decay fungi.

The water absorption decreased and the biological durability increased with samples that had been coated with wood oil before the tests.

In this study, significant differences between the properties of thermally modified sapwood and heartwood of pine were detected in water and decay exposure. The differences between the sapwood and heartwood of spruce were notably smaller. The modification temperature had a remarkable effect on the properties of wood; this effect was not linear in every case however.

As concluded, the wood species, sapwood and heartwood portions, and thermal modification temperature obviously have an influence on the biological and physical properties of thermally modified wood. These factors should be taken into account in production processes and applications as well as in testing.

Sini Metsä-Kortelainen. Differences between sapwood and heartwood of thermally modified Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*) under water and decay exposure [Lämpökäsitellyn kuusen (*Picea abies*) ja männyn (*Pinus sylvestris*) pinta- ja sydänpuun käyttäytyminen vesi- ja lahorasituksessa]. Espoo 2011. VTT Publications 771. 58 s. + liitt. 64 s.

**Avainsanat** 

decay, contact angle, heartwood, Norway spruce, sapwood, Scots pine, thermal modification, water absorption

#### Tiivistelmä

Puun lämpökäsittelymenetelmiä on kehitetty kosteuselämisen vähentämiseksi ja biologisen kestävyyden parantamiseksi. Tämän tutkimuksen tavoitteena oli selvittää lämpökäsitellyn männyn (*Pinus sylvestris*) ja kuusen (*Picea abies*) pintaja sydänpuun eroja kosteus- ja lahorasituksessa. Myös lämpökäsittelylämpötilan ja puun pintakäsittelyn vaikutusta tutkittiin.

Useita kokeita tehtiin sekä laboratoriossa että koekentällä kolmella toisiaan täydentävällä tutkimusmateriaalilla. Useimmissa kokeissa käytetty tutkimusmateriaali koostui neljässä eri lämpötilassa (170 °C, 190 °C, 210 °C ja 230 °C) käsitellystä männyn ja kuusen pinta- ja sydänpuusta. Vertailumateriaalina oli kokeesta riippuen käsittelemätöntä männyn ja kuusen pinta- ja sydänpuuta, lehtikuusta, bangkiraita, jättiläistuijaa, merbauta sekä painekyllästettyjä puumateriaaleja.

Lämpökäsittely vähensi kuusen pinta- ja sydänpuun vedenimeytymistä käsittelylämpötilasta riippuen kellutuskokeessa. Männyn pinta- ja sydänpuulla puolestaan vedenimeytyminen kellutuskokeessa joko kasvoi tai väheni eri lämpötiloissa tehdyn käsittelyn seurauksena. Männyn pintapuu imi vettä runsaammin ja nopeammin kuin muut puumateriaalit, kun taas männyn sydänpuu imi itseensä kaikkein vähiten vettä. Kontaktikulmamittauksessa vasta korkeimmassa 230 °C:n lämpötilassa käsitellyt puumateriaalit eivät olleet yhtä herkkiä imemään vettä itseensä kuin käsittelemättömät vertailumateriaalit.

Lämpökäsiteltyjen puumateriaalien lahonkestoa tutkittiin laboratoriossa ruskolahokokeella, jossa käytettiin kahta inkubaatioaikaa (6 ja 10 viikkoa) sekä kahta sientä (*Coniophora puteana* ja *Poria placenta*). Laboratoriossa tehtiin myös 32 viikon pituinen multalaatikkokoe pääasiassa katkolahottajien vaikutuksen tutkimiseksi. Koekentällä tarkasteltiin lämpökäsittelyn vaikutusta puun lahonkestoon maan pinnan yläpuolella toteutetussa lap-joint-kokeessa 1, 2, ja 9 vuoden rasituksen jälkeen. Lämpökäsittely paransi yleisesti kaikkien puumateriaalien lahonkestoa. Mitä korkeampi oli käsittelylämpötila, sitä enemmän lahonkesto parani. Lahonkeston kasvussa oli kuitenkin merkittäviä eroja eri materiaalien välillä. Multalaatikkokokeessa tarvittiin lahonkestoluokkien "kestävä" tai "erittäin kestävä" saavuttamiseksi lämpökäsittely katkolahoa vastaan kaikkein korkeimmassa 230 °C:n lämpötilassa. Ruskolahokoe antoi yleisesti hieman parempia tuloksia, mikä tarkoittaa, että lämpökäsittely jo alemmissa (190–210 °C) lämpötiloissa paransi etenkin männyn lahonkestoa merkittävästi. Kenttäkokeen tuloksilla yhdeksän vuoden rasituksen jälkeen oli hyvä korrelaatio laboratoriossa tehdyn ruskolahokokeen tulosten kanssa.

Lämpökäsittelyn sekä lahotuksen vaikutusta puun taivutuslujuuteen tutkittiin pienillä koekappaleilla. Sekä lämpökäsittely itse että lahotus alensivat puun lujuutta. Lahotuksen vaikutus oli kuitenkin huomattavasti merkittävämpi. Johtopäätöksenä voidaan todeta, että käsittelemätön puutavara on lujempaa kuin lämpökäsitelty puu, kunnes puumateriaali altistuu lahottajille.

Puun pintakäsittely vähensi selvästi veden imeytymistä kellutuskokeessa sekä paransi lahonkestoa ruskolahokokeessa kaikilla materiaaleilla.

Tässä tutkimuksessa havaittiin merkittäviä eroja kosteus- ja lahorasituksessa lämpökäsitellyn männyn pinta- ja sydänpuun välillä. Erot kuusen pinta- ja sydänpuun välillä olivat huomattavasti pienemmät. Myös käsittelylämpötila vaikutti merkittävästi puun ominaisuuksiin, joskin on huomattava, ettei lämpötilan vaikutus ollut lineaarista kaikissa tapauksissa.

Yhteenvetona voidaan todeta, että puulaji, pinta- ja sydänpuun osuudet sekä lämpökäsittelylämpötila vaikuttavat merkittävästi lämpöpuun biologisiin ja fysikaalisiin ominaisuuksiin. Nämä tekijät tulee ottaa huomioon tuotantoprosesseissa, käyttökohteissa sekä myös testauksessa.

#### **Academic dissertation**

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#### **Preface**

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Espoo, September 2011

Sini Metsä-Kortelainen

## List of publications

This thesis is based on the following publications, which are referred to in the text by Roman numerals I–VI:

- I Metsä-Kortelainen, S., Antikainen, T. & Viitaniemi, P. (2006). The water absorption of sapwood and heartwood of Scots pine and Norway spruce heat-treated at 170°C, 190°C, 210°C and 230°C. *Holz als Roh- und Werkstoff*, 64:3, 192–197.
- II Viitanen, H., Metsä-Kortelainen, S. & Laakso, T. (2006). Resistance of pine and spruce heartwood against decay The effect of wood chemical composition and coating with water-borne wood oil product (Doc. No. IRG/WP 06-10597). International Research Group on Wood Preservation.
- III Metsä-Kortelainen, S. & Viitanen, H. (2009). Decay resistance of sapwood and heartwood of untreated and thermally modified Scots pine and Norway spruce compared with some other wood species. *Wood Material Science and Engineering*, 4(3–4), 105–114.
- IV Metsä-Kortelainen, S. & Viitanen, H. (2010). Effect of fungal exposure on the strength of thermally modified Norway spruce and Scots pine. *Wood Material Science and Engineering*, 5(1), 13–23.
- V Metsä-Kortelainen, S. & Viitanen, H. (2011). Wettability of sapwood and heartwood of thermally modified Norway spruce and Scots pine. Published online in *European Journal of Wood and Wood Products* on 3 February 2011.
- VI Metsä-Kortelainen, S., Paajanen, L. & Viitanen, H. (2011). Durability of thermally modified Norway spruce and Scots pine in above ground conditions. Published online in *Wood Material Science and Engineering* on 26 April 2011.

## Author's contribution to the publications:

- I Sini Metsä-Kortelainen planned the experiments, performed the thermal modifications and water absorption test, analysed the data with Toni Antikainen and was responsible for writing the manuscript.
- II Sini Metsä-Kortelainen drew up the research plan and wrote the manuscript in close co-operation with the co-authors. Tapio Laakso was responsible for the chemical analysis and Sini Metsä-Kortelainen for the other tests and data analysis in co-operation with Hannu Viitanen.
- III, IV, V, VI Sini Metsä-Kortelainen drew up the research plan, analysed the data and was responsible for writing the manuscripts.

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## List of abbreviations

CCA copper chromium arsenic

EMC equilibrium moisture content

MC moisture content

MOE modulus of elasticity

MOR modulus of rupture

RH relative humidity

TBTO tributyl tin oxide

WRC Western red cedar

#### 1. Introduction

As a renewable and natural material, wood is widely used in construction. Many wood species are susceptible to weathering and fungal decay however. The resistance of wood to decay organisms has traditionally been improved with impregnation treatments using toxic chemicals. Increasing environmental pressures in the last decades have led to the development of new environmentally friendly methods for wood protection. Thermal modification, among other modification methods, is an alternative process for improving the properties of wood.

Different methods for thermal modification of wood have been developed in, among other places, Finland, France, the Netherlands and Germany. The common objective of these methods is to improve the dimensional stability and biological durability of wood without adding chemicals or biocides to the wood. Some of these processes are still under development, and others are already in commercial production. The Finnish ThermoWood® process is one of these thermal modification processes used in commercial production. The process is based on heating the wood material for a few hours at high temperatures above 180°C under normal pressure while protecting it with water vapour. This technology is the final result of long-term and persistent research and development work carried out at VTT since 1992.

In Finland, the first thermally modified wood producers started production at the end of the 1990s. In the beginning, the marketing of a completely new wood product was quite difficult, and the manufacturing processes also needed some enhancement. There were also problems with the quality of the thermally modified wood because of variations in the raw material, differences between modification processes and an inadequate quality control system. Thermally modified wood was also used in the wrong applications because consumers did not understand the behaviour and demands of the new product: the directions given to consumers were inadequate. The situation was improved over the period of a

few years however. One reason for the improvement was the establishment of the International Thermowood Association in December 2000. The association promotes the use of thermally modified wood, and production quality control, product classification and R&D activities are also important duties of the organisation. The method of thermal modification that was developed and patented at VTT has been licensed to members of the International Thermowood Association. Only members of the association are allowed to use of the trade name ThermoWood®.

Scots pine (*Pinus sylvestris*) and Norway spruce (*Picea abies*) are the main wood species used for industrial-scale thermal modification, though birch, aspen, alder and other wood species are also thermally modified in Finland. The production has grown year on year, and the main market area for ThermoWood® is currently the EU. Thermally modified wood is used in many applications that require enhanced dimensional stability and biological durability. The brown colour of the thermally modified wood is also seen as a benefit by the furniture industry. There are many good examples of thermally modified timber being used in different applications, such as exterior cladding, decking, flooring, garden furniture, panelling, kitchen furnishing and the interiors of bathrooms and saunas.

Thermal modification changes the chemical, physical and biological properties of wood. The colour of the wood material changes to brown and, at the same time, different degrees of weight loss of the wood are experienced. Thermal modification also improves the dimensional stability and biological durability of wood, though the bending and tensile strength decrease in relation to the intensity of the thermal modification. The reaction mechanisms and properties of thermally modified wood have been widely studied in many research institutes all over the world. Differences between thermally modified sapwood and heartwood have been reported less frequently, however, though the different chemical compositions and physical properties of sapwood and heartwood of many wood species are well known.

The aim of this thesis was to investigate the differences between the sapwood and heartwood of thermally modified Norway spruce and Scots pine under water and decay exposure. The effect of the temperature of the thermal modification process was also examined.

### 2. Background

#### 2.1 Properties of untreated Norway spruce and Scots pine

#### 2.1.1 Chemical composition

Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*) are both softwood species whose structures are anisotropic, consisting of different cell types, all of which have their own function. Over 90% of the cells of spruce and pine are tracheids, which are long and slender and have mechanical and conducting functions in the tree. Spruce and pine also consist of small amounts of parenchyma and epithelial cells, which have both storage and conduction functions. The water transport between adjacent cells occurs through pits. Growth rings of the wood material are composed of earlywood and latewood sections. The cell walls of latewood are thicker and the cell diameters smaller than those of earlywood (Fengel & Wegener, 1984). Due to the anisotropic structure, the physical properties of the wood material are different in the tangential, radial and longitudinal directions.

The main building materials of the wood cells are cellulose, hemicelluloses and lignin. Approximately 40–45% of the dry weight of most softwood species is cellulose (Fengel & Wegener, 1984; Sjöström, 1993). Cellulose is therefore the main constituent of trees and it forms a skeleton that is surrounded by other substances that function as a matrix. Cellulose gives the cell wall its strength, while hemicelluloses together with the lignin regulate the water content in the cell wall. The content of hemicelluloses and lignin in softwoods is typically approximately 20–30% and 25–30%, respectively (Sjöström, 1993). Cellulose and hemicelluloses are hydrophilic but lignin is hydrophobic.

Wood material also contains small amounts of extractives that protect the wood against biodeterioration and insect attacks. Extractives are wood compo-

nents that can be extracted by means of polar or non-polar solvents and are most characteristically found in large quantities in the heartwood (see Section 2.1.2) but also in the resin canals of conifers or as reserve materials in the living portion of the wood (Fengel & Wegener, 1984; Sjöström, 1993). The composition of extractives varies widely from species to species, and the amount of extractives varies even between different parts of the same tree.

#### 2.1.2 Sapwood and heartwood

At a certain age, the inner wood of the stem of most trees begins to change into completely dead heartwood. The dying cells produce heartwood extractives from the nutrients in them whilst the bordered pits are aspirated. The sapwood gives structural support to the living tree, acts as a food storage reservoir and transports water. The heartwood consists of dead cells, and the extractives in heartwood protect the tree against biological attack, lower the water content and act as fungicides (Wise & Jahn, 1952).

The chemical composition and physical properties of the heartwood of many wood species vary and differ from those of sapwood. In many wood species, the heartwood is darker than the sapwood or becomes darker during use. The content and composition of the extractives in heartwood fluctuate. The heartwood of many wood species is naturally more resistant to attack by decay organisms than the sapwood. The weight, density, hygroscopicity and permeability of heartwood and sapwood have been reported to be quite different (Kollmann & Côté, 1968; Kärkkäinen, 2003). There is wide variation in the amount of heartwood between different wood species, individual stems and different parts of a single stem. The age and growth rate of the tree affect the content and composition of extractives, and the amount of heartwood is higher in older trees than in younger ones, though it decreases from the butt to the top of the tree (Saarelainen, 1981).

The heartwood of Scots pine is darker than the sapwood, but there is no distinct colour difference between the heartwood and the sapwood of Norway spruce. The moisture content of living pine and spruce heartwood is much lower than that of sapwood. The permeability of heartwood has also been reported to be much lower than that of sapwood because of the aspiration of the bordered pits. The difference between the permeability of heartwood and sapwood has been observed to be greater in pine than spruce (Elowson et al., 2003; Kärkkäinen, 2003).

#### 2.1.3 Natural durability

Wood can be attacked by different kinds of fungi and bacteria when the prevailing relative humidity and temperature are high enough. Brown- and white-rot fungi mainly belong to a subdivision of *Basidiomycetes*. Most brown-rot fungi prefer to attack softwoods, while white-rot fungi attack hardwoods. Soft-rot fungi are found in softwoods and hardwoods, and they belong to *Ascomycetes* and *Fungi imperfecti* as well as blue-stain and mould fungi, which together are wood discolouring fungi (Fengel & Wegener, 1984). Brown-rot fungi degrade the cellulose and hemicelluloses of wood while white-rot fungi mainly degrade the lignin and cellulose. Soft-rot fungi destroy the cellulose in the most important parts of the cell wall. Wood loses its strength as a consequence of the degradation of the wood components, especially the cellulose, caused by the decay fungi (Saarelainen, 1981).

The natural durability, which is defined as 'the inherent resistance of wood to attack by wood destroying organisms' according to EN 350-1 (1994), depends on the wood species. The natural durability of the Finnish wood species is limited and the durability of the heartwood of the most important economic timbers are classified as 'moderately durable' or 'slightly durable' (Scots pine) and 'slightly durable' (Norway spruce) according to EN 350-2 (1994). The durability of the sapwood of Finnish softwoods is even more limited than that of heartwood.

Heartwood extractives are the principal source of low water permeability and increased natural durability or decay resistance (Olsson et al., 2001). The lower fungal durability of sapwood may, on some occasions, be a consequence of its greater permeability (Kollmann & Côté, 1968). The most important factors affecting the increased natural decay resistance of Scots pine heartwood are the concentration of total phenolics and certain stilbenes like pinosylvin and its monomethyl ether (Harju et al., 2003; Venäläinen et al., 2003).

#### 2.2 Thermal modification processes

There are several thermal modification methods for wood (Homan & Jorissen, 2004). These have the common aim of increasing the durability of wood against decay organisms and to decrease the swelling and shrinking of wood by subjecting the wood to temperatures between 150°C and 250°C, in an atmosphere with a low oxygen content. Short descriptions of the most common industrial-scale

processes for thermal modification of wood are presented in Sections 2.2.1–2.2.5.

#### 2.2.1 ThermoWood® process

The Finnish ThermoWood® process is based on heating the wood material for a few hours at temperatures over 180°C at atmospheric pressure using water vapour (Viitaniemi, 1997a; Jämsä & Viitaniemi, 2001). The water vapour creates a protective atmosphere to prevent the wood from burning and cracking. Either green or kiln-dried wood material can be used. The process can be divided into three phases: drying, thermal modification and conditioning.

#### 2.2.2 PLATO

The PLATO process in the Netherlands principally consists of two stages, hydrothermolysis and dry curing, with an intermediate drying operation (Homan et al., 2000; Militz & Tjeerdsma, 2001). The wood species and the thickness and form of the timber have an effect on the process time needed, and green or airdried wood can be used.

#### 2.2.3 Retification and Le Bois Perdure

In France, two modification processes are in use. The first one is referred to as Retification, in which normally dried wood is heated up to 210–240°C in a nitrogen atmosphere. The second French process, Le Bois Perdure, allows for the use of fresh wood. In this process, the fresh wood is first dried and then heated up to 230°C in a steam atmosphere. The steam is generated from the water in the wood (Vernois, 2001).

#### 2.2.4 Menz Holz

The Menz Holz process in Germany is based on heating the wood at temperatures of between 180°C and 260°C in a hot vegetable oil bath (Rapp & Sailer, 2001).

#### 2.2.5 WTT Thermo treatment

The WTT Thermo treatment process developed in Denmark is a new technology in which the wood material is treated in a pressurised steam atmosphere at 3–19 bars and working temperatures of 140–210°C. The process uses pre-dried wood and does not fully dry the wood material during the thermo treatment (WTT, 2011).

# 2.3 The effects of thermal modification on the properties of wood

Thermal modification changes the chemical composition of wood, thereby altering the appearance and physical and biological properties of the wood. Several factors influence the properties of thermally modified wood: wood species, sapwood / heartwood, dimension or size of the timber/specimen, moisture content, thermal modification method used and, in particular, the intensity of the thermal modification, which usually depends on the thermal modification temperature and time (Mitchell, 1988). The higher the thermal modification temperature and the longer the treatment time, the more significant the changes in the wood properties are. The effects of thermal modification on wood material have been widely studied and all of the factors mentioned above related to the modification process have to be taken into account when examining the results.

Thermally modified wood is like a new wood species and its special characteristics have to be taken into account during the whole production process. Generally, thermally modified wood is more susceptible to mechanical damage during further processing than normal dried wood. Painting and gluing also have to be carried out very carefully using process parameters optimised for thermally modified wood. Good joining requires care because thermally modified wood is quite brittle and may also contain residual acids that can cause corrosion on fasteners (Jermer & Andersson, 2005). Thermally modified wood also needs a UV-protective coating because it turns grey like normal wood when exposed to weather or UV light (Jämsä et al., 2000; Miklečić et al., 2011).

#### 2.3.1 Chemical properties

Wood is a complex, composite material consisting mainly of cellulose, hemicelluloses, lignin and extractives (Fengel & Wegener, 1984; Sjöström, 1993).

Thermal modification changes the chemical structure of wood. Alén et al. (2002) concluded that the main structural components (cellulose, hemicelluloses and lignin) of Norway spruce wood, particularly hemicelluloses, were converted into volatiles and other pyrolysis products. Sivonen et al. (2002) studied thermally modified Scots pine with NMR spectroscopy and detected an increase in the relative crystallinity of cellulose and the destruction and deacetylation of hemicelluloses. In a study by Andersson et al. (2005), an increment in the mass fraction of crystalline cellulose was perceived, and an increase in the porosity of the cell wall was also observed using X-ray scattering methods. Nuopponen et al. (2003) studied the behaviour of the extractives of Scots pine during thermal modification and concluded that the resin acids in the radial resin canals moved to the surface of wood at temperatures of between 100°C and 180°C and disappeared from the wood surface at higher temperatures. Degradation or modification of hemicelluloses, amorphous cellulose and lignin, and evaporation and polymerisation of extractives during thermal modification have also been reported by Tjeerdsma et al. (1998), Viitaniemi et al. (2002), Weiland and Guyonnet (2003), Wikberg and Maunu (2004), Tjeerdsma and Militz (2005), Boonstra and Tjeerdsma (2006), Mburu et al. (2006), Esteves et al. (2008a) and González-Peña et al. (2009).

#### 2.3.2 Physical properties

Wood turns brown and loses weight as a consequence of the degradation and evaporation of wood components during thermal modification (Viitaniemi & Jämsä, 1996; Bekhta & Niemz, 2003; Mohebby & Sanaei, 2005; Johansson & Morén, 2006; Esteves et al., 2007a). The weight loss during thermal modification depends mainly on the modification temperature and time, and it has a strong relationship to many wood properties (Zaman et al., 2000; Welzbacher et al., 2007). The colour of thermally modified wood also correlates with the intensity of the thermal modification (Brischke et al., 2007; Schnabel et al., 2007; Esteves et al., 2008b; González-Peña & Hale, 2009a, 2009b).

Thermal modification reduces the shrinking and swelling of wood, with thermally modified wood being more stable than normal wood in conditions of changing humidity (Virta, 2005; Tuong & Li, 2011). Thermal modification reduces the equilibrium moisture content and in most cases also the water absorption and wettability of wood (Viitaniemi & Jämsä, 1996; Kamdem at al., 2002; Bekhta & Niemz, 2003; Pétrissans et al., 2003; Hakkou et al., 2005a; Popper et

al., 2005; Repellin & Guyonnet, 2005; Wang & Cooper, 2005; Awoyemi, 2006; Follrich et al., 2006; Kartal et al., 2007; Kocaefe et al., 2008; Korkut & Bektaş, 2008; Almeida et al., 2009; Herajärvi, 2009; Ohmae et al., 2009). The principal factor for enhanced dimensional stability and reduced water absorption is likely to be the reduction in the number of hydroxyl groups of the hemicelluloses (Weiland & Guyonnet, 2003; Boonstra & Tjeerdsma, 2006). Moreover, Hakkou et al. (2005b) suggested that plasticisation of lignin, leading to a reorganisation of the lignocellulosic polymeric components of wood, could explain the reduced wettability of thermally modified wood.

As a result of thermal modification, wood becomes more brittle, and the bending, tensile and compression strength decrease in relation to the intensity of the thermal modification and wood species (Viitaniemi & Jämsä, 1996; Viitaniemi, 1997b; Santos, 2000; Kubojima et al., 2000; Bekhta & Niemz, 2003; Unsal & Ayrilmis, 2005; Poncsák et al., 2006; Yildiz et al., 2006; Boonstra et al., 2007a; Esteves et al., 2007b; Shi et al., 2007; Korkut et al., 2008; Gunduz et al., 2009; Majano-Majano et al., 2010). Sundqvist et al. (2006) suggested that the decrease in mechanical properties is related to acid formation during the modification process. Phuong et al. (2007) proposed that the brittleness of the thermally modified wood was a consequence of the degradation of amorphous polysaccharides.

#### 2.3.3 Biological properties

The improved fungal durability of thermally modified wood has been reported in a considerable number of publications (Viitanen et al., 1994; Viitaniemi & Jämsä, 1996; Sailer et al., 2000; Tjeerdsma et al., 2000; Kamdem et al., 2002; Gosselink et al., 2004; Hale et al., 2005; Welzbacher & Rapp, 2005; Boonstra et al., 2007b; Mburu et al., 2007; Welzbacher & Rapp, 2007). There may be several reasons for the increased resistance to fungal attack. During thermal modification, the wood becomes more hydrophobic, which limits the absorption of water and may suppress fungal growth. Thermal modification changes the chemical composition of wood, making it more difficult for fungi to attack the wood material, and it may generate new extractives, which may have fungicidal or fungistatic effects (Kamdem et al., 2000; Kotilainen, 2000; Sivonen et al., 2003; Weiland & Guynnoet, 2003; Mburu et al., 2006). The most plausible hypothesis for the durability improvement is the chemical modification and degradation of wood during thermal modification according to Hakkou et al. (2006) and Lekounougou et al. (2009). Thermally modified wood has also been noted to be

#### 2. Background

less susceptible to discolouring organisms, e.g., mould and blue stain than untreated wood (Viitaniemi & Jämsä, 1996; Edlund & Jermer, 2004; Petrič et al., 2006; Kocaefe et al., 2007; Frühwald et al., 2008).

#### 3. Material and methods

The resistance of thermally modified sapwood and heartwood of Scots pine and Norway spruce to decay and water exposure was investigated with several laboratory and field tests that supported and complemented each other. The research material was based on the same wood batch in all the laboratory tests except for the experiments relation to wood coatings. The results are therefore truly comparable. A schematic representation of the wood materials of the main batch, the thermal modifications and the tests performed is shown in Figure 1. The materials and test methods are described briefly below and in more detailed in Papers I–VI.

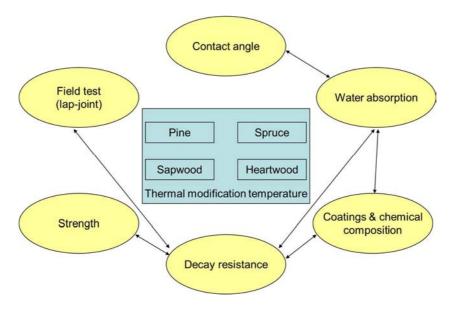


Figure 1. Schematic description of the wood materials of the main batch, the thermal modifications and the performed tests.

#### 3.1 Research materials

Three separate research materials (1–3) were used in the tests and are presented in the following sections.

# 3.1.1 Research material (1) used in laboratory tests with thermally modified wood

Industrially kiln-dried sapwood and heartwood planks of Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*) were selected from a sawmill situated in SE Finland. For the laboratory thermal modification operations, 18 planks of each test material with only small variations in density and widths of year rings were selected. The planks were as clear sapwood or heartwood as possible. All the 4.8-m-long planks were planed, split down the middle and cut into four 1.2-m-long pieces. One half of the planks were left as reference material and the other half were thermally modified (Figure 2). A more detailed description of the raw material selection is given in Paper I. This test material was also used in the studies presented in Papers III–V.

In addition, industrially kiln-dried Siberian larch, merbau, bangkirai and Western red cedar (WRC) were chosen as reference material for the decay and strength tests.

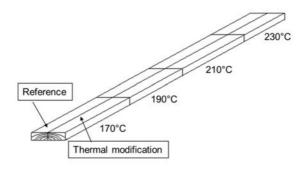


Figure 2. Cutting of the test planks for the thermal modifications.

# 3.1.2 Research material (2) used in laboratory tests with unmodified wood

Norway spruce and Scots pine logs were sawn from trees felled from four different locations in the southern parts of Finland. The logs were sawn into planks,

which consisted, as far as possible, of either sapwood or heartwood. The planks were then carefully dried at a temperature of 60°C. One half of the samples prepared from this research material were coated throughout with a water-based primer and pigmented wood oil and the other half were left as an uncoated reference material. The extractive content of the research material was analysed by determining the methanol extract profile. The analysis method and the selection of raw material are explained in detail in Paper II and by Viitanen et al. (2006).

# 3.1.3 Research material (3) used in field test with thermally modified wood

The field test was started two years before the other experiments for which industrially kiln-dried Norway spruce and Scots pine planks produced in south-eastern Finland (50 x 100 mm<sup>2</sup>) were selected. Scots pine impregnated with tributyl tin oxide (TBTO) and copper, chromium and arsenic (CCA) was also selected as a reference material according to Paper VI. Sorting between the sapwood and heartwood was not performed.

#### 3.2 Thermal modifications

The ThermoWood® method was used in all the thermal modification operations. The temperature inside the wood and the atmosphere in the kiln were measured during the processes. The thermal modification operations were controlled with these measured temperatures. The thermal modification time at the target temperature was 3 hours in every test run while the thermal modification temperature was changed. The test planks were conditioned carefully under steam immediately after the thermal modification operations.

#### 3.2.1 Thermal modifications for the laboratory tests

The small 1.2-m-long planks from research material 1 described in Section 3.1.1 were thermally modified in a laboratory kiln at VTT. There were four different wood materials: pine sapwood, pine heartwood, spruce sapwood and spruce heartwood, and four different thermal modification temperatures: 170°C, 190°C, 210°C and 230°C, and 16 test runs were therefore carried out in total. Temperatures of 190°C and 210°C were selected to correspond to temperatures commonly used in industrial thermal modification processes (Thermo-S 190°C, Thermo-

D 212°C) and temperatures of 170°C and 230°C were included to gather additional information on the behaviour of the wood material. The thermal modification operations are presented fully in Paper I.

#### 3.2.2 Thermal modifications for the field test

The thermal modifications of research material 3 described in Section 3.1.3 were performed under accurately controlled conditions at the YTI Research Centre in Mikkeli, Finland. The planks chosen for modification were treated at temperatures of 195°C and 210°C.

#### 3.3 Methods

Several standard tests or tests based on standards were performed in the laboratory and field. Three separate research materials (Sections 3.1.1–3.1.3) were used in the tests according to Figure 3. These tests, described briefly below, were selected to give systematic information about the behaviour of the thermally modified wood products in experiments in relation to moisture and decay.

#### Scots pine and Norway spruce research materials

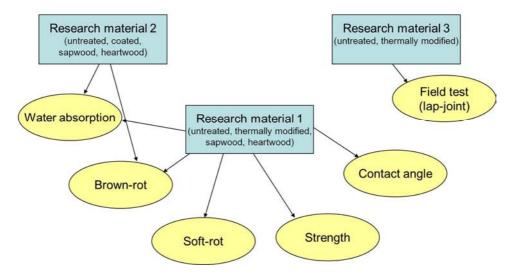


Figure 3. Research materials 1–3 and different tests performed in this study.

#### 3.3.1 Water absorption

Two water absorption tests were performed (Papers I–II), and the test procedure based on the standard EN 927-5 (2000) was almost the same in both tests. The first test was performed with thermally modified research material 1 presented in Sections 3.1.1 and 3.2.1. Ten replicate specimens of each wood material (pine sapwood, pine heartwood, spruce sapwood, spruce heartwood, five different temperatures) were sealed and floated outer face downwards in a water basin. The moisture content (MC) of the specimens was determined after 6, 23, 45, 71 and 146 hours of flotation.

The second water absorption test was performed with test material 2 described in Section 3.1.2. The effect of wood coating on water absorption was studied. Three samples per test material were prepared and the specimens were floated in water for 72 hours and the water uptake calculated.

#### 3.3.2 Wettability

The wettability of thermally modified sapwood and heartwood of Scots pine and Norway spruce (the raw material and thermal modifications are summarised in Sections 3.1.1 and 3.2.1) was assessed by measuring the static contact angles of distilled water on the surfaces as a function of time. The specimens were conditioned at 65% relative humidity (RH) and 20°C to a constant mass before the measurements were taken in the earlywood area perpendicular to the axis of the wood grain. The test procedure is presented in more detail in Paper V.

#### 3.3.3 Decay resistance

The decay resistance of thermally modified, unmodified and coated sapwood and heartwood of Scots pine and Norway spruce was examined in the laboratory. A field test in above-ground conditions was also performed with Scots pine and Norway spruce material with mixed portions of sapwood and heartwood.

#### 3.3.3.1 Decay test against brown-rot fungi

A decay test against brown-rot fungi was performed using two different test materials according to a mini-decay test (Bravery, 1979). The sample size was  $5 \times 20 \times 35 \text{ mm}^3$  and the incubation times were 6 and 10 weeks in both tests. In

the first test, thermally modified sapwood and heartwood of Scots pine and Norway spruce (research material 1 described in Sections 3.1.1 and 3.2.1) were exposed to two different fungi *Coniophora puteana* and *Poria placenta*. After incubation, the moisture content and weight loss were analysed and the results classified into durability classes based on the median mass losses according to CEN/TS 15083-1 (2005). The details of the decay test are presented in Paper III.

The effect of a coating on the fungal resistance of unmodified sapwood and heartwood of Scots pine and Norway spruce (test material 2 described in Section 3.1.2) to *Coniophora puteana* was studied in the second test. The test is described in more detail in Paper II.

#### 3.3.3.2 Decay test against soft-rot fungi

The natural durability of thermally modified sapwood and heartwood of Norway spruce and Scots pine and of the other reference wood species was determined according to CEN/TS 15083-2 (2005). The specimens (5 x 10 x 100 mm³) were sawn from the same research material 1 used in the brown-rot test with two different fungi (Sections 3.1.1 and 3.2.1). The modulus of elasticity (MOE) was determined in a static three-point bending test before and after the test (Figure 4) without breaking the specimens. The incubation time was 32 weeks, after which the mass loss was also determined. The results of the soft-rot test were classified into durability classes according to CEN/TS 15083-2 (2005). The test and the measurement of the MOE values are presented in Paper III.



Figure 4. The static bending test, central loading method.

#### 3.3.3.3 Lap-joint field test

Thermally modified and unmodified Scots pine and Norway spruce samples were prepared for a horizontal lap-joint test according to ENV 12037 (1996). Two different types of impregnated wood were also selected as reference material. Raw material 3 described in Sections 3.1.3 and 3.2.2 was used. The samples were installed on exposure racks at the Otaniemi test site in southern Finland in November 2001 and the discoloration and decay were inspected separately after 1, 2 and 9 years. The test is described in detail in Paper VI.

#### 3.3.4 Bending strength

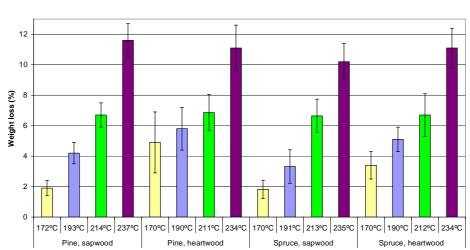
The effect of fungal exposure on the strength properties of thermally modified sapwood and heartwood of Norway spruce and Scots pine was measured in a static bending test. Half of the specimens originated from the 32-week decay test against soft-rot fungi and half of the specimens were unexposed wood material from raw material 1 described in Sections 3.1.1 and 3.2.1. All of the specimens were conditioned at 65% relative humidity (RH) and 20°C to constant mass before the bending test, after which the MOE and the modulus of the rupture (MOR) were calculated. A more itemised description of the test is given in Paper IV.

#### 4. Results and discussion

#### 4.1 Weight loss after thermal modification

The weight loss correlates with the many properties of thermally modified wood, as described in a publication by Viitaniemi (1997b). As weight loss is dependent on several factors, some variables were eliminated, and for this reason, the thermal modification time was 3 hours in every test run and the size of the test planks was also constant. The density variation within the planks of each test material was also small. In this study, the intensity of the thermal modifications was thereby only dependent on the thermal modification temperature, wood species and wood part (sapwood/heartwood).

Viitaniemi and Jämsä (1996) detected that as a consequence of degradation and evaporation of wood components during thermal modification, wood loses weight. In this study, the weight loss was calculated and presented as a function of wood materials and actual treatment temperatures, as shown in Figure 5. The calculation of the weight loss is explained in Paper I. The actual modification temperatures are average values of the highest temperatures measured from inside the wood over 3 hours. The target temperatures (170°C, 190°C, 210°C and 230°C) were slightly exceeded, particularly with the highest thermal modification temperatures. In general, the weight loss correlated strongly with the thermal modification temperature. The weight loss of pine heartwood at the lower modification temperatures of 170–190°C was notably higher, however, which was probably a consequence of the evaporation of extractives during modification (Nuopponen et al., 2003). The weight loss of spruce heartwood at these two lower temperatures was also more significant than that of spruce sapwood.



#### Weight losses of wood materials in thermal modifications at actual temperatures

Figure 5. Weight losses and actual temperatures in thermal modification operations.

#### 4.2 Thermally modified wood under water exposure

#### 4.2.1 Water absorption

The water absorption differences between the sapwood and heartwood of Scots pine and Norway spruce were studied as a function of time. The results are presented in Paper I (Figures 4–5). The water absorption properties are connected to the shrinking and swelling of wood as well as the biological durability aspects.

The water absorption of the spruce materials correlated strongly with the thermal modification temperature: the higher the treatment temperature, the lower the moisture content. The moisture content of the spruce materials increased evenly as a function of time. A similar connection between thermal modification temperature and moisture content was not detected with pine materials, which, on the other hand, exhibited a clear correlation between the wood part (sapwood/heartwood) and the moisture content. In fact, the water absorption differences between the sapwood and heartwood of pine were significant. The moisture content of the untreated pine sapwood samples was more than double after 71 hours of floating compared with the heartwood samples. Thermal modification mainly decreased the water absorption of pine heartwood. On the other hand, thermal modification of pine sapwood at temperatures of 170–210°C in-

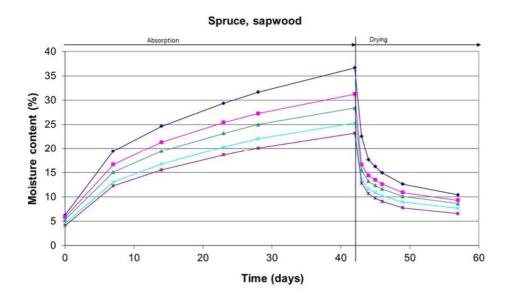
creased the water absorption compared with the untreated reference samples. Kartal et al. (2007) measured the water absorption of sugi (*Cryptomeria japonica* D. Don) thermally modified at temperatures of 180°C and 220°C for 2 and 4 hours and detected a similar increase in moisture content with samples thermally modified in milder conditions (180°C, 2 hours). In another publication (Mohebby & Sanaei, 2005), the water absorption of thermally modified beech (*Fagus orientalis* Lipsky) at a temperature of 160°C was decreased but increased after thermal modification at 180°C compared with unmodified samples. A thorough explanation for this interesting increase in water absorption of thermally modified wood at lower temperatures has not been given in the papers referred to. Evidently, this phenomenon should be studied further in the future.

In this study, the moisture content differences between the reference and thermally modified pine sapwood materials were also more significant at the early stages of the test than the situation after approximately six days of floating.

#### **Extended water absorption**

The water absorption test was extended after the results of Paper I were published because the water absorption differences between untreated and thermally modified materials seemed to decrease as a function of the floating time. The specimens were floated again in a water basin for 42 days, after which the specimens were left to dry at room temperature for 15 days and the moisture content evaluated by weighing the samples at short intervals.

The results of the extended water absorption and drying test are presented in Figures 6–7. Note that the scale of the y-axis of the pine sapwood differs from the scale used in the other figures.



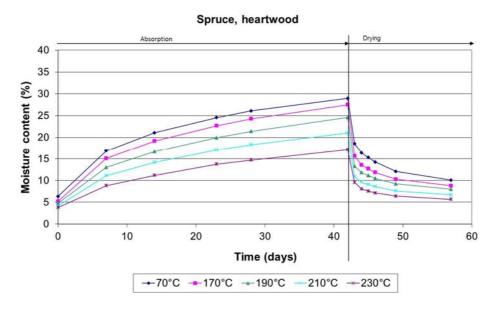
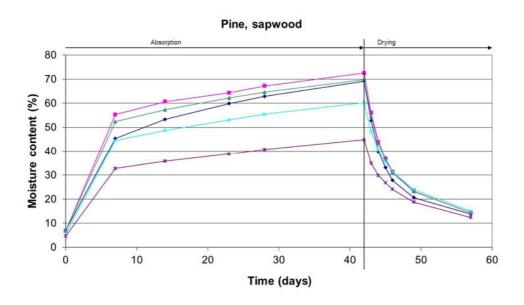


Figure 6. Extended water absorption of spruce sapwood and heartwood.

#### 4. Results and discussion



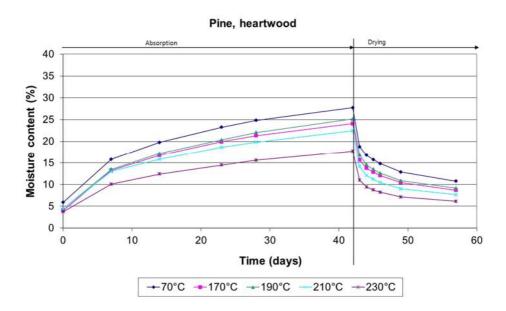


Figure 7. Extended water absorption of pine sapwood and heartwood.

The differences between the sapwood and heartwood of spruce were quite small in the extended water absorption test. The level of moisture content of the spruce heartwood was lower throughout than that of the sapwood, however, which agrees with the findings of Bergström and Blom (2005). Thermal modification decreased the moisture content values quite linearly.

The moisture contents of the pine heartwood were rather similar to the moisture contents of the spruce heartwood. The water absorption of pine heartwood had a negligible correlation with the thermal modification temperature however. In fact, the behaviour of the thermally modified pine heartwood seemed very interesting as it absorbed water less after thermal modification at a very low temperature of 170°C, it then became more hydrophilic in the temperature range 190–210°C until thermal modification at a very high temperature of 230°C made it more resistant to water again.

The sapwood of pine had a completely different behaviour compared with the other wood materials in the test. All the pine sapwood specimens reached fairly high moisture contents after floating for 7 days. Even the moisture contents of specimens thermally modified at 230°C were over 30%. Thermal modification at temperatures of 170–190°C increased water absorption compared with the unmodified reference samples, and these differences were also evident in the drying phase of the test. In fact, the drying of the pine sapwood was surprisingly slow compared with the rate of moistening. In practice, this means that pine sapwood remains more wetted and for longer periods than the other wood materials during moisture exposure, which may expose the wood material to a greater risk of biological attack.

## Effect of coating

In the third water absorption test, untreated sapwood and heartwood of pine, and sapwood of spruce were coated with water-based primer and wood oil and floated for 72 hours (Paper II, Figure 3). The water uptake of the untreated spruce and pine heartwood samples was nearly at the same level, which was very much lower than that of the pine sapwood. The wood coating significantly decreased the water uptake of all the wood materials. For example, the water uptake of the coated pine sapwood decreased to the level of untreated pine heartwood.

## 4.2.2 Wettability

A decrease in the wettability of thermally modified wood measured by contact angle analysis has been reported in several publications (Pétrissans et al., 2003; Esteves et al., 2007b; Follrich et al., 2006; Kocaefe et al., 2008). Whether the

#### 4. Results and discussion

material under investigation was sapwood or heartwood has not generally been taken into account. Oliveira et al. (2010), however, studied the wettability of the sapwood and heartwood of *Araucaria angustifolia* thermally modified at several temperatures and detected a significant increase in contact angle values except in thermally modified heartwood, the value of which decreased drastically at 200°C. The authors suggested that enhanced wettability may partly have been a consequence of the drying cracks detected from the surface of the samples.

In this study (Paper V), the differences in contact angle between the sapwood and heartwood of thermally modified Scots pine and Norway spruce were examined. The other aim was to find possible connections with the results of the floating test. The results after two different measurement times (1.1 s and 25.3 s) are presented in Figures 8–9. In general, the wettability of the sapwood of pine was higher than that of the pine heartwood, as expected. Thermal modification decreased or increased water repellency, however, depending on the thermal modification temperature, wood part and measurement time. The water repellency of pine sapwood was only increased for the material that had been thermally modified at a temperature of 230°C at the early stages of the test. Thermal modification apparently slowed down water absorption and spreading, since after 25 seconds only the samples that had been thermally modified at a temperature of 170°C were recorded as being less repellent than the untreated reference samples. Similar differences in the absorption rates as a function of time were not detected with pine heartwood samples. Thermal modification increased the wettability of pine heartwood except for the samples that had been thermally modified at a temperature of 170°C. It is very interesting that thermal modification of pine at a temperature of 170°C resulted in the sapwood being less repellent and the heartwood more repellent. This observation and mainly the other findings in the contact angle tests with pine materials are in line with the results of the water absorption test.

The trends of the contact angle curves of the sapwood and heartwood of spruce were quite similar: only thermal modification at temperature of 230°C increased the water repellency. The contact angles of the spruce sapwood were higher than those of heartwood on some occasions, which was unexpected. These results are not in line with the observations of the floating test, in which the thermal modification reduced the water absorption of spruce materials almost linearly.

The contact angle results were compared with the weight losses that took place during the thermal modification operations. Based on these results, it can be generalised that a weight loss of 10% or more is needed to reduce the wettability of the research material, except in the case of the pine heartwood thermally modified at 170°C. Other possible factors affecting the wettability changes are the migration of the extractives or resin acids to the surface of the wood during ageing (especially in the case of the untreated materials) and the modification and complicated degradation of the wood components during thermal modification. These factors have been discussed in more detailed in Paper V. It is obvious that the wetting properties of thermally modified wood depend significantly on the wood species, wood part and thermal modification temperature.

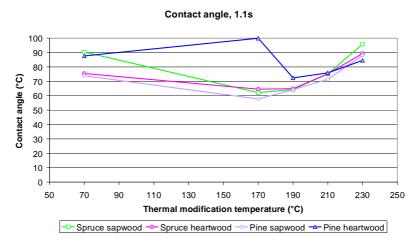


Figure 8. Contact angles of wood materials recorded after 1.1 seconds.

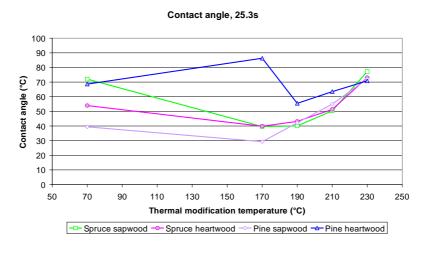


Figure 9. Contact angles of wood materials recorded after 25.3 seconds.

## 4.3 Thermally modified wood under decay exposure

## 4.3.1 Decay resistance against brown-rot fungi

The biological durability of thermally modified sapwood and heartwood of Norway spruce and Scots pine was examined with small samples in the laboratory. The research material was exposed to brown-rot fungi *Coniophora puteana* and *Poria placenta* as shown in Figure 10.





Figure 10. Brown-rot test samples, Poria placenta (left) and Coniophora puteana (right).

The heartwood of untreated pine was the most durable wood material, while the differences between untreated sapwood and heartwood of spruce and sapwood of pine were quite small after the brown-rot test (Paper III, Figures 7–10). *Poria placenta* attacked the untreated wood materials more rapidly; the Coniophora puteana fungus, however, caused mass losses that were the same or higher than *Poria placenta* after 10 weeks' exposure.

The thermal modification increased the durability of all the wood materials, which is in agreement with previous studies (Esteves & Pereira, 2009). Thermal modification increased the durability against *Coniophora puteana* in particular, though the resistance to degradation by *Poria placenta* was also improved, especially with the pine materials. Boonstra et al. (2007b) detected similar differences in the durability of thermally modified wood materials against different types of brown-rot fungi.

The durability of the wood materials improved with increasing thermal modification temperatures. On average, the untreated wood materials were classified into the natural durability class 'slightly durable' which is in line with the standard EN 350, parts 1 and 2 (1994), and after thermal modification at a temperature of 230°C, the same materials were classified as 'very durable'. The thermal modification at temperatures of 190°C and 210°C, which correspond to the temperatures that are commonly used in industrial ThermoWood processes, increased the biological durability against *Coniophora puteana* slightly and significantly, respectively. The resistance of spruce materials against *Poria placenta*, especially after an incubation time of 10 weeks, was not significantly improved until thermal modification at the highest temperature (230°C).

## 4.3.1.1 Moisture content of the samples

A high enough moisture content (25–30%) is needed to ensure fungal activity in wood. The equilibrium moisture contents (EMC) before and after, and the moisture content (MC) after the brown-rot tests with *Coniophora puteana* and *Poria placenta* are presented in Tables 1–2. The moisture contents after testing the wood materials were 30% or more except for the samples thermally modified at 230°C in the test with *Poria placenta*. The brown-rot tests were therefore relevant, and fungal activity had been assured at least in most of the cases based on these moisture content values.

The equilibrium moisture contents (RH 65%, 20°C) of the wood samples were lower after the brown-rot test compared with the values before the test. The differences between the equilibrium moisture contents before and after the test were more significant in samples that had been thermally modified at lower temperatures under 210°C. In fact, the moisture content of the samples treated at higher temperatures reached the same or even higher equilibrium moisture contents after the test than before it. Brown-rot fungi mainly degrade the polysaccharides of the softwoods (Fengel & Wegener, 1984). It seems that for this reason, the biodegraded wood material reaches lower moisture contents as the moisture mainly binds to the wood material by the polysaccharides. The samples treated at higher temperatures were not significantly biodegraded, and the equilibrium moisture contents were thus not reduced.

#### 4. Results and discussion

Table 1. Equilibrium moisture content before and after, and moisture content after 6 and 10 weeks of brown-rot tests with *Coniophora puteana*. The standard deviation is in parentheses.

		6 weeks			10 weeks		
		MC (%)	MC (%)	MC (%)	MC (%)	MC (%)	MC (%)
		RH65%	RH65%		RH65%	RH65%	
		Before test	After test	After test	Before test	After test	After test
Spruce, sapwood	untreated	10.6 (0.2)	7.7 (0.3)	53.9 (2.8)	10.6 (0.2)	7.3 (0.2)	71.9 (11.3)
	170°C	9.0 (0.4)	7.6 (0.2)	48.7 (4.6)	8.8 (0.3)	7.0 (0.2)	57.2 (6.9)
	190°C	8.1 (0.4)	7.4 (0.2)	44.0 (16.9)	8.2 (0.7)	6.7 (0.2)	42.6 (9.3)
	210°C	7.0 (0.5)	6.8 (0.2)	34.0 (6.0)	6.9 (0.6)	6.5 (0.2)	28.6 (3.7)
	230°C	5.9 (0.6)	6.4 (0.2)	42.7 (6.6)	6.1 (0.6)	5.9 (0.2)	46.2 (19.3)
Spruce, heartwood	untreated	10.3 (0.2)	7.7 (0.1)	54.3 (3.3)	9.7 (1.0)	7.3 (0.4)	68.3 (8.1)
	170°C	8.0 (0.7)	7.6 (0.1)	48.7 (12.6)	8.2 (0.6)	7.4 (0.1)	53.7 (11.8)
	190°C	8.0 (0.4)	7.2 (0.2)	33.9 (4.3)	7.9 (0.3)	6.9 (0.3)	41.0 (6.2)
	210°C	6.1 (0.6)	6.8 (0.3)	39.9 (13.1)	6.0 (0.7)	6.6 (0.3)	41.0 (13.8)
	230°C	5.3 (0.7)	6.1 (0.3)	48.5 (11.3)	5.2 (0.6)	5.7 (0.4)	37.9 (13.7)
Pine, sapwood	untreated	10.1 (0.2)	7.8 (0.1)	55.9 (5.3)	10.2 (0.2)	7.2 (0.2)	64.3 (5.4)
	170°C	8.8 (0.2)	7.5 (0.1)	49.3 (4.0)	8.9 (0.2)	7.1 (0.2)	59.5 (8.6)
	190°C	8.4 (2.3)	7.4 (0.2)	44.6 (4.2)	7.8 (0.4)	6.7 (0.2)	44.4 (2.9)
	210°C	6.7 (0.6)	7.0 (0.1)	31.0 (4.8)	6.5 (0.7)	6.5 (0.3)	45.5 (23.5)
	230°C	5.4 (0.5)	6.5 (0.2)	49.3 (33.1)	5.5 (0.6)	5.9 (0.2)	57.9 (31.3)
Pine, heartwood	untreated	9.8 (0.2)	7.4 (0.2)	45.5 (6.9)	9.8 (0.2)	6.7 (0.3)	57.6 (6.5)
	170°C	7.4 (0.8)	7.1 (0.1)	38.4 (3.9)	7.4 (0.8)	6.7 (0.2)	46.4 (5.5)
	190°C	6.7 (0.4)	7.2 (0.3)	33.5 (2.5)	6.6 (0.3)	6.9 (0.2)	39.1 (5.3)
	210°C	6.4 (0.5)	7.0 (0.1)	32.2 (5.8)	6.1 (0.4)	6.6 (0.1)	30.2 (7.1)
	230°C	4.7 (0.4)	6.2 (0.2)	40.8 (13.0)	4.8 (0.6)	5.9 (0.2)	38.4 (18.7)

Table 2. Equilibrium moisture content before and after, and moisture content after 6 and 10 weeks of the brown-rot test with *Poria placenta*. The standard deviation is in parentheses.

			6 weeks			10 weeks	
		MC (%)	MC (%)	MC (%)	MC (%)	MC (%)	MC (%)
		RH65%	RH65%		RH65%	RH65%	
		Before test	After test	After test	Before test	After test	After test
Spruce, sapwood	untreated	10.6 (0.2)	7.2 (0.1)	58.7 (4.2)	10.6 (0.0)	7.3 (0.3)	68.8 (12.0)
	170°C	8.9 (0.3)	7.4 (0.3)	64.9 (11.1)	9.0 (0.3)	7.1 (0.2)	60.2 (6.2)
	190°C	8.4 (0.4)	7.0 (0.1)	50.0 (8.4)	8.0 (0.6)	6.9 (0.2)	47.6 (5.4)
	210°C	6.8 (0.5)	6.9 (0.2)	31.0 (6.1)	7.0 (0.5)	6.7 (0.3)	40.3 (9.5)
	230°C	5.7 (0.6)	6.3 (0.3)	27.4 (4.4)	5.6 (0.6)	5.7 (0.3)	17.9 (2.7)
Spruce, heartwood	untreated	10.1 (0.3)	7.4 (0.2)	59.7 (7.5)	10.1 (0.2)	7.4 (0.1)	67.0 (10.3)
	170°C	7.2 (1.1)	7.3 (0.3)	52.4 (10.7)	7.3 (1.1)	7.3 (0.2)	54.0 (6.4)
	190°C	8.0 (0.3)	7.2 (0.1)	44.6 (6.5)	7.8 (0.3)	6.9 (0.0)	54.7 (15.7)
	210°C	6.2 (0.6)	6.5 (0.2)	35.3 (12.1)	6.4 (0.7)	6.5 (0.2)	36.4 (9.2)
	230°C	5.2 (0.8)	5.9 (0.2)	37.4 (14.7)	5.3 (0.7)	5.7 (0.2)	22.6 (1.4)
Pine, sapwood	untreated	10.2 (0.1)	7.4 (0.1)	57.1 (2.6)	10.2 (0.1)	7.3 (0.1)	74.9 (15.8)
	170°C	8.7 (0.2)	7.1 (0.1)	55.6 (7.1)	8.8 (0.3)	7.2 (0.2)	63.1 (4.5)
	190°C	7.9 (0.3)	7.1 (0.1)	58.3 (7.7)	7.9 (0.5)	6.9 (0.3)	60.1 (9.8)
	210°C	6.8 (0.6)	6.7 (0.2)	29.7 (2.6)	6.8 (0.6)	6.6 (0.2)	31.5 (12.9)
	230°C	4.9 (0.8)	6.2 (0.2)	24.4 (4.4)	5.2 (0.9)	5.8 (0.4)	28.9 (5.7)
Pine, heartwood	untreated	9.6 (0.2)	7.2 (0.3)	59.4 (6.8)	9.8 (0.2)	7.1 (0.3)	56.0 (21.8)
	170°C	6.9 (0.5)	7.1 (0.3)	52.8 (9.6)	6.9 (0.5)	6.5 (0.2)	55.9 (6.5)
	190°C	7.0 (0.5)	7.0 (0.2)	42.9 (2.1)	6.7 (0.4)	6.8 (0.2)	57.3 (2.7)
	210°C	6.4 (0.6)	6.7 (0.1)	28.6 (4.5)	6.1 (0.6)	6.5 (0.3)	27.3 (10.1)
	230°C	5.0 (0.4)	6.2 (0.2)	22.4 (2.6)	5.1 (0.3)	5.8 (0.3)	19.5 (1.9)

## 4.3.1.2 Effect of the wood's chemical composition and coating on decay resistance

The second durability test against *Coniophora puteana* was performed with untreated and coated sapwood and heartwood of Scots pine and Norway spruce (Paper II, Figures 4–5). The effect of the chemical composition on the biological durability was studied.

The differences in mass loss as well as in extractive content values between the sapwood and heartwood were significantly smaller with spruce than with pine. The coating slowed down the fungal activity and significantly increased the biological durability of all the samples especially after 6 weeks' exposure.

There was a very high variation among the mass losses of untreated pine heartwood in contrast with the other wood materials after both exposure times. The chemical composition and, especially the pinosylvin content of pine heartwood, also had a great deal of variation within the different samples sawn out from different logs. This observation is in agreement with, e.g., Bergström et al. (1999). A strong correlation between the pinosylvin content and mass loss in the decay test was detected. The samples with high pinosylvin content were, on average, more durable.

## 4.3.2 Decay resistance against soft-rot fungi

The natural durability of thermally modified sapwood and heartwood and four other reference wood species was determined with small specimens inserted into containers with unsterile soil for 32 weeks (Figure 11). The results are presented in more detailed in Paper III, Figures 1–5.

In general, there was a similar trend in the mass and modulus of elasticity (MOE) loss values, and the correlation between these values was very high. The MOE loss values were much greater than the mass loss values, however, which indicates that MOE is a more sensitive measure for detecting fungal attack in the wood. This is in agreement with Humar et al. (2006) and Temiz and Yilziz (2006).

Due to the high MOE losses in the decay test, the untreated sapwood and heartwood of pine and spruce were all classified into the worst durability class 'not durable' together with Western red cedar (WRC), which was one of the reference wood species. Larch and bangkirai were classified into the 'slightly durable' class and merbau into the class 'moderately durable'. Basically, all the

wood species reached worse durability classes in this test compared with the classes specified in the standard EN 350, parts 1 and 2 (1994). This indicates test conditions that were very or even too harsh, and it is worth taking into account that the soft-rot test was originally intended to measure the resistance of wood preservatives against soft-rot fungi.



Figure 11. The specimens were incubated in containers in the soft rot test.

The mass and MOE loss differences between the sapwood and heartwood of pine were again more evident. Thermal modification increased the biological durability in all of the cases, but rather high temperatures (210–230°C) were needed to influence the durability class. Both spruce materials were classified into the durability class 'durable' and the sapwood and heartwood pine reached the classes 'moderately durable' and 'very durable', respectively, after thermal modification at a temperature of 230°C.

## Strength of decayed wood

Fairly high thermal modification temperatures were needed to increase the biological durability of wood materials in the soft-rot test. As is well known, thermal modification decreases the strength properties of wood in relation to the thermal modification temperature and time. The mechanical properties are also

reduced by fungal exposure. The strength of decayed, thermally modified wood was therefore investigated in the static bending strength test, which is presented in more detailed in Paper IV.

On average, the thermal modification and decay exposure both decreased the MOE and the bending strength (modulus of rupture, MOR) values. The effect of fungal exposure on strength was more significant than the effect of the thermal modification itself. The MOE of the undecayed samples was reduced slightly less than the MOR as a consequence of thermal modification. A decrease in the MOE of thermally modified wood was likely, partly due to the density loss in the thermal modification operations because the density usually correlates linearly with the MOE. The effect of the thermal modification temperature on the MOE of the decayed samples was more significant than the effect on the MOR.

The MOE and MOR values were reduced slightly more with spruce than with pine in a comparison between undecayed reference samples and thermally modified samples. On average, the loss in the mechanical properties of undecayed sapwood and heartwood of pine and heartwood of spruce was not significant until thermal modification at a temperature of 230°C. The mechanical properties of spruce sapwood was most affected: thermal modification at 210°C reduced the MOE and MOR by 15% and with samples thermally modified at 230°C the MOR was reduced by almost 30%.

Thermal modification increased the MOE and MOR of decayed wood material compared with the unmodified samples in almost every case: the higher the modification temperature, the higher the MOE and MOR values.

The MOE and MOR loss values caused by the fungal exposure were determined. The decrease in strength and stiffness was greater with untreated wood than with thermally modified samples. The decrease in mechanical properties was connected to the thermal modification temperature: the higher the temperature, the less the strength was reduced by the fungal attack. Thermal modification of both heartwood materials at 230°C seemed to provide protection against fungal exposure especially on the MOE. Once again, the differences between the sapwood and heartwood of pine were more evident than with spruce.

It can be concluded that unmodified wood material will be stronger than thermally modified wood material until wood is exposed to decay fungi. Thus, the selection of the thermal modification temperature is a compromise between improved fungal durability and reduced mechanical properties, which should be taken into account in design and be in accordance with the application demands.

## 4.3.3 Decay resistance in above-ground conditions

Thermally modified Scots pine and Norway spruce were subjected to decay exposure in above-ground conditions in the field. The results of the lap-joint are described in detail in Paper VI (Figures 1–3).

Discoloration started from the upper sides of the wood samples, which were full of blue stain, dirt and natural greying after one year of exposure. During the subsequent years, the discoloration spread to the bottom sides and joint areas of the samples. Thermal modification decreased in accordance with the modification temperature, the discoloration on the bottom sides and, especially, in the joint areas after 9 years of exposure, which is in line with Edlund and Jermer (2004).

The first signs of decay were detected in untreated pine after 2 years in the field and, after 9 years of exposure, most of these samples reached the failure rating, as well as many of the untreated spruce specimens. Thermal modification increased the biological durability of both wood species significantly. In general, the pine was attacked slightly more than the spruce. The results of the lap-joint field test correlated quite well with the results of the laboratory test with brownrot fungi (Section 4.3.1). This indicates that a quick and simple laboratory test may give preliminary results of the behaviour and durability of wooden material to outdoor exposure.

## 5. Conclusions

The main conclusions that can be drawn from this study:

- The differences between the main properties of sapwood and heartwood are significantly greater with pine than with spruce.
- Pine heartwood is the most resistant and pine sapwood the most susceptible wood material to water and decay exposure.
- Thermal modification of sapwood and heartwood of spruce, and heartwood of pine decrease water absorption compared with untreated wood.
- Thermal modification of pine sapwood at temperatures of 170–210°C increases water absorption compared with unmodified wood.
- A relatively high thermal modification temperature (230°C) is needed to decrease the wettability of wood measured as contact angles.
- Thermal modification improves the fungal durability of pine and spruce materials both in laboratory and field tests. In general, the higher the temperature the more the durability increases.
- The fungal attack in the soft-rot test reduces the mechanical properties of wood more than the thermal modification itself.
- Wood coatings effectively decrease the water absorption and increase the fungal durability of wood materials.

The wood species, sapwood and heartwood portions, and the thermal modification temperature obviously have an influence on the biological and physical properties of thermally modified wood. It is highly presumable that the type of the thermal modification process also has a significant effect on the final result. The properties of thermally modified wood, according to the demands of the end-use application, can be tailored more precisely in the modification process when the nature of the raw material is taken into account.

It is notable that the effect of the thermal modification intensity on the properties of the wood is not linear in every case. For example, thermal modification reduces the water absorption of sapwood and heartwood of spruce and heartwood of pine. The water absorption of thermally modified pine sapwood is increased in some cases however. On the other hand, sapwood and heartwood of spruce need a higher modification temperature than pine materials to improve significantly the biological durability against *Poria placenta*.

It is important to use thermally modified wood only in conditions in which moisture and decay stresses are not too high. The moisture and decay stresses of different kinds of wood materials are tested according to standardised tests and expressed as, e.g., durability classes. It is still quite difficult, however, to use the durability classes and other test results in defining the right use classes according to EN 335-1 (2006) and in predicting the service life of wood products. The present test methods should be developed to give a better understanding of the behaviour of wood products in different use conditions, not only in the worst case situations. For example, in a laboratory test with different decay fungi, several exposure times could be used to correspond with conditions in different applications.

Thermally modified wood is like a new wood species and its special characteristics should be taken into account in different production processes and applications as well as in testing. The term 'thermally modified wood' should not be used without also specifying the thermal modification process, wood species and, if possible, the wood part, especially if the properties of the sapwood and heartwood of the wood species concerned differ remarkably from each other. It is notable that the properties of the original wood material have an important effect on the properties of the wood material after thermal modification.

It is likely to be challenging to take into account the special properties of different kinds of wood materials in the production line. The good quality and desired properties of thermally modified wood can be achieved by strict sorting and grading of the raw material, careful controlling of the thermal modification process and precise quality control of the end product however. The thermal modification intensity should be the same for every single board in the same batch and the effect of the thermal modification should be measured afterwards from the modified samples. When the result of the thermal modification is thoroughly known, then it is possible to choose the right kind of thermally modified wood according to the application demands.

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## **PUBLICATION II**

## Resistance of pine and spruce heartwood against decay – The effect of wood chemical composition and coating with water-borne wood oil product

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## THE INTERNATIONAL RESEARCH GROUP ON WOOD PROTECTION

Section 1 Biology

## Resistance of pine and spruce heartwood against decay

# The effect of wood chemical composition and coating with water-borne wood oil product

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## Resistance of pine and spruce heartwood against decay

# The effect of wood chemical composition and coating with water-borne wood oil product

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#### **ABSTRACT**

Natural durability of wood has been widely studied, but the combination of the natural durability and different treatments has not been the focus of many studies. The durability of wooden products is mainly based on the water permeability and the resistance against organisms. In this study, the water absorption and decay resistance of sapwood and heartwood of Scots pine and Norway spruce were examined. The effects of the wood origin and the coating with water-borne wood oil were also observed. The wood oil is penetrating treatment which is recommended for decking and garden furniture. The results were compared to the chemical composition of wood. The water absorption of untreated pine sapwood was significantly higher than that of pine heartwood or spruce. Decay resistance of pine heartwood was relatively high. However, the decay resistance was widely varied among different pine heartwood samples and in some cases this variation was significantly higher than that of pine sapwood or spruce. The effect of wood oil coating on decay resistance of pine heartwood was more significant than on pine sapwood. Elevated decay resistance was also found among coated spruce samples, but no significant difference between sap and heartwood was found. Decay resistance was comparable with water permeability and pinosylvin content of wood.

**Keywords:** Brown rot, Chemical composition, Decay resistance, Durability, Pine, Spruce, Water absorption, Wood oil treatment.

## 1. INTRODUCTION

According to the European standard EN 350-1, 2 (1994) based on laboratory and field test results using different wood species and according to experiences of using different wood material as a building material, heartwood is naturally more durable than sapwood. The better natural durability of heartwood in many wood species is mainly originated from the wood microstructure and chemical composition (Rayner and Boddy 1988). In many wood species, however, wide variation has been found between individual trees and within the individual logs. The variation of durability or decay resistance has been compensated by using chemical treatments, preservation, modifications or different kinds of surface treatments of wood (Zabel and Morrell 1992).

The heartwood of Scots pine (*Pinus sylvestris*) is classified to natural durability classes 3 - 4 (moderately to slightly durable) and the heartwood Norway spruce (*Picea abies*) belongs to the natural durability class 4 (slightly durable) according to the standard EN 350-2 (1994).

Discussion about the durability of wooden products and service life has been activated, and the exploitation of the natural durable wood material is in focus. However, there are problems concerning the using of results of biological tests for service life prediction of wooden products. There are several methods for testing the biological durability of impregnated wood material, but very few of them are suitable for testing the durability of wooden products (CEN 2005).

The natural durability of wood has been widely studied, but the combination of the natural durability and different treatments has been less studied. In this study, the water permeability and the decay resistance of coated wood against brown rot fungus were determined by using modified standard tests. Also main chemical characteristic for wood biological durability were studied.

#### 2. MATERIALS AND METHODS

The sample logs of Scots pine (Pinus sylvestris) and Norway spruce (Picea abies) were sawn from different trees felled from the southern parts of Finland. The origin of trees is mentioned by using codes. The trees were from four different places: Pine trees were from Puumala (U) and Ruokolahti (R) and spruce trees were from Parikkala (P) and Heinola (H). Logs from the trees are numbered using tree code (first number) and log code (last number, 0 = butt log, 1 = middle  $\log_{10} 2 = \log_{10} \log_{10} 2$ . The  $\log_{10} 2 = \log_{10} \log_{10} 2$ . The  $\log_{10} 2 = \log_{10} \log_{10} 2$ . possible, and these planks were dried very carefully at temperature of 60°C. After drying and conditioning (RH65%, 20°C) to constant mass, test specimens were sawn out of the planks. For the mini decay test (Bravery 1979, Venäläinen et al. 2003), 10 replicate specimens per test material were sawn. Half of the specimens were sawn out from sapwood and half from heartwood planks, and half of the specimens were coated by dipping with water-borne primer and thereafter with water-borne pigmented wood oil. The dried matter contents of the primer and the wood oil were 16 vol-% and 11 vol-%, respectively. The wood oil is recommended to be used in wood decking and garden furniture. Pine specimens were collected from eight different logs and spruce specimens from six logs. The size of the specimen was 5 x 20 x 35 mm<sup>3</sup>. The samples were dried at 60°C for 48 hours, after which they were cooled in a desiccator and weighed to an accuracy of 1 mg. Then the samples were sterilised by using a radiation dose of between 25 kGy and 50 kGy of radioisotope <sup>60</sup>Co source. The samples were subjected to mini decay test on agar using Coniophora puteana (Bam Ebw 15) as a test organism. The incubation times were 6 and 10 weeks, and after incubation the moisture content and mass loss were analysed according to the standard EN 113 (1996).

The water uptake to the uncoated and coated samples was studied by using modified EN 927-5 (2000) method. The sample size was  $15 \times 40 \times 320 \text{ mm}^3$  and three samples were used per test material. Half of the samples were coated with a brush throughout with same primer and wood oil as the mini test samples. The specimens were conditioned to constant mass at RH65% and  $20^{\circ}$ C and then weighed. Then, the specimens were floated outer face downwards in a water basin and weighed again after 72 hours floating.

The extractive content of wood samples was analysed by determining the methanol extract profile. 50 mg finely powdered sapwood and heartwood samples were sonicated with 2 ml methanol (p.a.) for 30 min. 0,4 mg diethylstilbesterol (Sigma D 4628) and 0,2 mg m-erythritol (Fluka 45670) were used as internal standards. After centrifugation 1 ml extract solution was evaporated dry under nitrogen. Before GC/MS analysis, dried samples were silylated with 0,5 ml 20 % TMSI-pyridine mixture (TMSI=1-(trimethylsilyl)imidazole). The GC/MS analyses were performed using a HP 6890 GC-system equipped with mass selective detector 5873 and HP-5 capillary column (30m x 0,25 mm i.d., 0,25 um film thickness). Helium was used as carrier gas,

flow 1,5 ml/min. The chromatographic conditions were as follows: initially temperature 180°C; temperature rate, 5°C/min; final temperature 300°C for 5 min; injector temperature 280°C and split ratio 1:20. MS-interface temperature was 300°C and ion source temperature 230°C. Mass spectra were obtained by electron impact (EI mode) ionization energy 70 eV. Stilbenes (PS and PSM) and total resin acids were quantified from chromatograms using internal standards and pure reagents (Arbonova) for stilbenes and 75 % abietic acid (Fluka 00010) for resin acids.

#### 3. RESULTS

## 3.1 Uptake of wood oil, water uptake and wood chemical composition

The uptake and spreading rates of wood oil during dipping and brushing treatments are presented in the table 1. Results of chemical composition of wood are shown in the figures 1 and 2. Water uptake in the floating test (EN 927-5) is presented in the figure 3.

Table 1: Average results of spreading rate of primer and wood oil by dipping and brushing (g/m<sup>2</sup>).

_	Dipping rate f	for decay samples	Spreading rate for floating samples		
Wood Material	Water based	Water based	Water based	Water based	
	primer	pigmented wood oil	primer	pigmented wood oil	
Pine sapwood	129.7	75.8	81.5	55.9	
Pine heartwood	91.2	61.0	65.9	54.0	
Spruce sapwood	124.7	75.3	80.6	70.5	
Spruce heartwood	102.0	72.3	=	=	

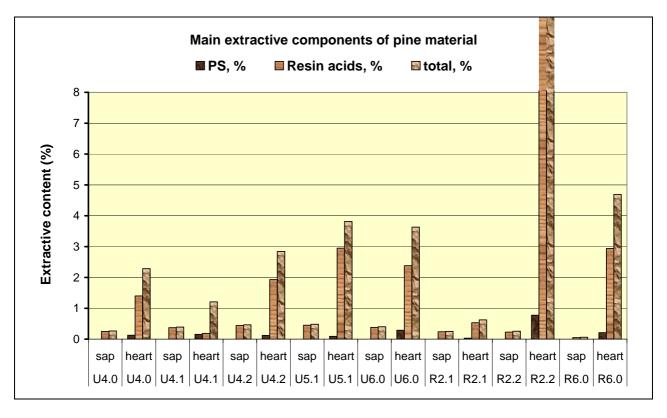


Figure 1. Analyse results of extractive contents (%) of Scots pine wood (pinosylvin, resin acids and total extractive content).

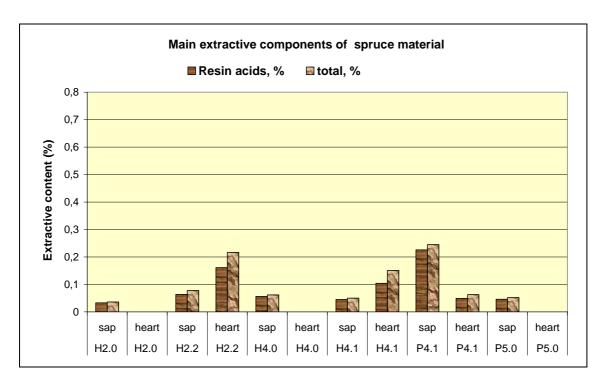


Figure 2. Analyse results of extractive contents (%) of Norway spruce wood (resin acids and total extractive content).



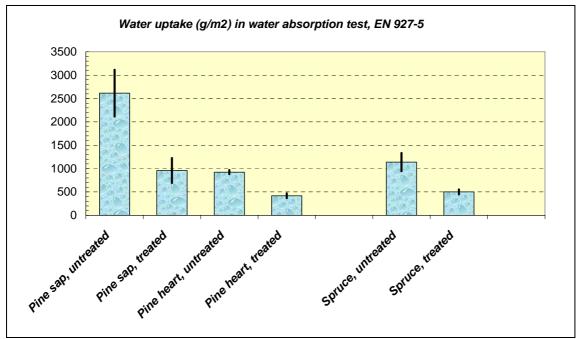


Figure 3. Water uptake of treated and untreated pine and spruce in a modified EN 927-5 test. Average results of different trees and logs.

The average water uptake of the untreated pine sapwood samples was high, which means high water permeability. The water uptake of wood oil coated pine sapwood was significant lower, as well as that of un-treated pine heartwood and untreated spruce. The water uptake was very low into the wood oil coated pine heartwood and spruce samples. The samples were not weathered prior to the water permeability test.

## 3.2 Decay resistance

Mass losses of untreated and treated pine and spruce sapwood are shown in the figures 4 and 5. The origin of wood material is mentioned by using short codes.

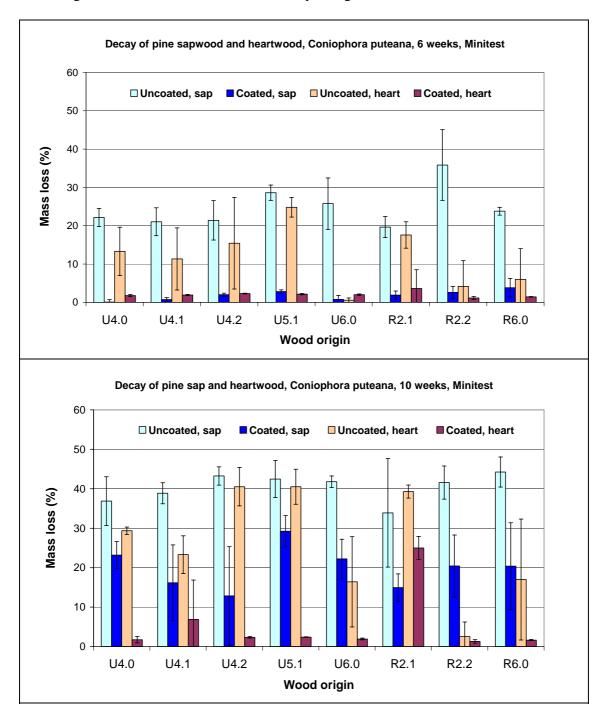


Figure 4: Mass loss of untreated and treated pine sap and heartwood after incubation of 6 and 10 weeks in decay test with *Coniophora puteana* on agar.

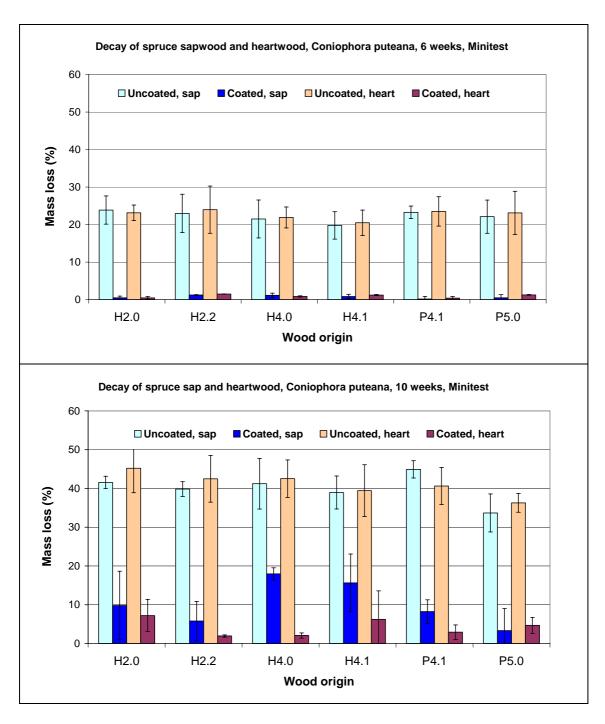


Figure 5: Mass loss of untreated and treated spruce sap and heartwood after incubation of 6 and 10 weeks in decay test with *Coniophora puteana* on agar.

The results of decay resistance in the mini test using *Coniophora puteana* correlated with the water permeability (Figure 6), but also with the wood chemical composition, especially with pinosylvin content of pine heartwood (Figure 7). The average mass loss of untreated pine sapwood after exposure of 6 weeks varied between 20 and 35 % and after 10 weeks between 35 and 45 %. There was a very high variation among mass losses of untreated pine heartwood after both exposure times. After 6 weeks, the lowest average mass loss of pine heartwood was around 4 % and the highest around 25 %, and the mass loss of samples treated with wood oil was very low for both sap and heartwood. After 10 weeks exposure, the mass losses of all untreated

heartwood samples were higher than after 6 weeks, but samples from one log (R2.2) showed especially low mass loss reflecting high a extractive content. However, the mass loss of the samples from R2.1 (from the same tree as R2.2) was significantly higher reflecting a very low extractive content of wood. The results of the chemical analyses of the heartwood sample R 2.1 in the figure 1 show a relatively low extractive content, which may refer to possibility of having sapwood or equal than sapwood in this heartwood sample.

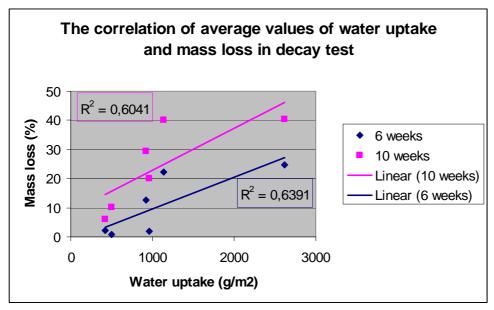


Figure 6. The correlation of results of water absorption and decay tests.

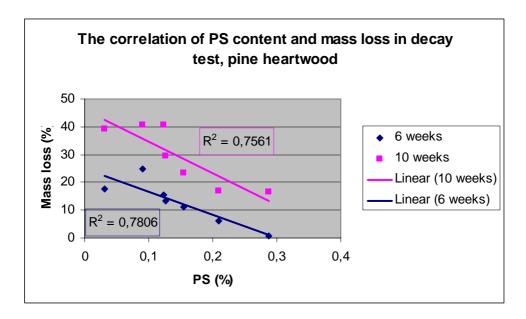


Figure 7. The correlation of pinosylvin content and mass loss in decay test, pine heartwood.

## 4. DISCUSSION

The results of the decay test of spruce differed notably from the results of pine. There was not any significant difference between mass loss of untreated spruce sap and heartwood, and the mass loss of spruce heartwood was significantly higher than that of pine heartwood. The mass loss of wood oil treated sapwood of spruce was significantly lower than that of untreated sapwood. Also, the decay resistance of wood oil treated spruce heartwood was in average around the same level than that of treated pine heartwood. Water permeability of spruce was significantly lower than that of pine sapwood. This may add the resistance and durability of spruce in use condition, where moisture stress is not too severe, e.g. claddings, fences (EN 335-1). The results of the present screening test showed, that coated and selected pine heartwood would be reliable material for wooden products also in high moisture exposure conditions (e.g. applications in use class 3 like decking and garden furniture in this case). Wide variation was found within the decay resistance of pine heartwood even among the limited test material used in the present study. Variation in decay resistance of pine heartwood has been found in many studies (van Acker et al. 1999, Harju et al. 2002, Venäläinen et al. 2003). In this study, pinosylvin seemed to be the most important chemical compounds for the decay resistance against brown rot. The effect of pinosylvin content on decay resistance has been found also in many other studies (e.g. Harju et al. 2003, Venäläinen et al. 2003).

The spruce could be an optional wood species for standardized tests for measuring resistance of coated wood products against decay, because the effect of the wood part (sapwood / heartwood) is not so important than with pine. This would be argued also due to the use of spruce as a material for coated building products. For screening tests of biological durability of wooden products, it would be useful to have several exposure time periods during a test. This would help to evaluate the effect of resistance of wooden products for preliminary evaluation of life time in different exposure conditions. Also water permeability tests should be included. In this context, the screening test system should be as convenient, reliable, fast and cheap as possible.

#### 5. CONCLUSIONS

The chemical composition of wood seemed to be an important factor effecting on the decay resistance of wood. Especially the pinosylvin content of pine heartwood was a significant factor. The difference of decay resistance between pine heartwood and spruce heartwood reflected very well with the chemical composition of the wood. Surface treatment using water-borne wood oil, however, affected on the water permeability and also on the decay resistance of wood. The samples were not preconditioned (weathered, leached or evaporated) prior to the exposure to decay fungus. In the next phase, the decay resistance after 4 and 10 months natural weathering will be studied.

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# **PUBLICATION III**

# Decay resistance of sapwood and heartwood of untreated and thermally modified Scots pine and Norway spruce compared with some other wood species

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# **ORIGINAL ARTICLE**

# Decay resistance of sapwood and heartwood of untreated and thermally modified Scots pine and Norway spruce compared with some other wood species

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#### Abstract

Thermal modification has been developed for an industrial method to increase the biological durability and dimensional stability of wood. In this study the effects of thermal modification on resistance against soft- and brown-rot fungi of sapwood and heartwood of Scots pine and Norway spruce were investigated using laboratory test methods. Natural durability against soft-rot microfungi was determined according to CEN/TS 15083-2 (2005) by measuring the mass loss and modulus of elasticity (MOE) loss after an incubation period of 32 weeks. An agar block test was used to determine the resistance to two brown-rot fungi using two exposure periods. In particular, the effect of the temperature of the thermal modification was studied, and the results were compared with results from untreated pine and spruce samples. The decay resistance of reference untreated wood species (Siberian larch, bangkirai, merbau and western red cedar) was also studied in the soft-rot test. On average, the soft-rot and brown-rot tests gave quite similar results. In general, the untreated heartwood of pine was more resistant to decay than the sapwood of pine and the sapwood and heartwood of spruce. Thermal modification increased the biological durability of all samples. The effect of thermal modification seemed to be most effective within pine heartwood. However, very high thermal modification temperature over 230°C was needed to reach resistance against decay comparable with the durability classes of "durable" or "very durable" in the soft-rot test. The brown-rot test gave slightly better durability classes than the soft-rot test. The most durable untreated wood species was merbau, the durability of which could be evaluated as equal to the durability class "moderately durable".

**Keywords:** Biological durability, brown rot, decay, heartwood, high temperature, Norway spruce, sapwood, Scots pine, soft rot, thermal modification.

# Introduction

Thermal modification at high temperatures, above 180°C, changes the chemical, biological and physical properties of wood. The effects of thermal modification on wood have been widely studied. According to the published results, hemicelluloses and amorphous cellulose are partly degraded or modified, the structure of lignin is modified and a part of extractives, particularly resin acids, is evaporated or modified (Kotilainen, 2000; Viitaniemi *et al.*, 2002; Wikberg, 2004; Nuopponen, 2005; Boonstra & Tjeerdsma, 2006; Mburu *et al.*, 2006). As a consequence of chemical changes in the wood material, the appearance and the biological and physical properties of the wood are also changed. The colour

turns to deep brown and the wood loses its weight (Viitaniemi & Jämsä, 1996; Viitaniemi, 1997a; Bekhta & Niemz, 2003). Thermal modification significantly improves the dimensional stability and reduces the equilibrium moisture content of wood, and, in most cases, water absorption is reduced, depending on the wood species (Viitaniemi, 1997b; Jämsä et al., 2000; Kamdem et al., 2002; Bekhta & Niemz, 2003; Pétrissans et al., 2003; Hakkou et al., 2005; Repellin & Guyonnet, 2005; Metsä-Kortelainen et al., 2006). However, the wood becomes more brittle and bending and tension strength decrease in relation to the level of thermal modification (Viitaniemi & Jämsä, 1996; Kamdem et al., 2002; Bekhta & Niemz, 2003).

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One of the main targets of thermal modification is to increase the biological durability of wood. Viitanen et al. (1994) reported that the decay resistance to brown-rot fungus Coniophora puteana according to EN 113 and soft-rot fungi according to prENV807 in thermally modified spruce was significantly improved, depending on the level of the thermal modification. Weiland and Guyonnet (2003) also used an EN 113 standard test and detected that resistance to fungal degradation in maritime pine and beech increased by 43% and 74%, respectively, compared with untreated wood. Hakkou et al. (2006) found an important correlation between the biological durability and temperature of thermal modification. Improved biological durability in different thermally modified wood species was also reported by Sailer et al. (2000), Kamdem et al. (2002), Edlund and Jermer (2004), Jones et al. (2006) and Mburu et al. (2006).

According to the published papers, there may be several reasons for the increased biological durability of thermally modified wood. During thermal modification the wood becomes more hydrophobic, which limits the absorption of water into the wood and may prevent fungal growth. Thermal modification changes the chemical composition of wood, making it more difficult for fungi to attack the wood material, and it may also generate new extractives, which may act as fungicides. Wood polymers may also be modified or degraded in thermal modification and for this reason the wood cannot be used as a nutritive source by decay fungi (Kotilainen, 2000; Weiland & Guynnoet, 2003).

It has long been known that the chemical composition and physical and biological properties of heartwood are different from those of the sapwood within many wood species. The sapwood of many softwoods, for instance, contains fewer extractives than the heartwood (Fengel & Wegener, 1984). A relatively high percentage of phenolic substances, pinosylvins and acetone-soluble extractives, particularly resin acids, protects the wood against microbiological attack (Sjöström, 1993; Harju *et al.*, 2003; Venäläinen *et al.*, 2003). The water permeability and hygroscopicity of heartwood are smaller than those of sapwood, and this may reduce the potential fungal growth and activity in wood (Kollmann & Côté, 1968).

Viitanen et al. (2006) studied the fungal resistance to C. puteana in the sapwood and heartwood of uncoated and coated Scots pine and Norway spruce. Significant differences were observed between the decay resistance of sapwood and heartwood, particularly within pine. The origin of the wood also strongly affected the decay resistance. Nuopponen (2005) discovered that thermal modification at high

temperatures (>200°C) removes extractives, particularly resin acids, from wood. Therefore, the amount of resin acids may not be the most critical factor for the decay resistance of thermally modified wood. The decay resistance of thermally modified heartwood has been less studied. The aim of this study was to determine the differences in decay resistance of sapwood and heartwood of Scots pine and Norway spruce thermally modified at different temperatures. The results were compared with the results for other wood species, such as Siberian larch, bangkirai, merbau and western red cedar (WRC).

## Materials and methods

Materials

Test planks of Scots pine (Pinus sylvestris) and Norway spruce (Picea abies) were selected from Finnish sawmills. The planks were industrially kilndried at a temperature of approximately 70°C to a moisture content of 11–15%. Half of the planks were sawn from the sapwood part of the logs and half from the heartwood. The selection criteria were the cleanness of the sapwood or heartwood, small variation in the year rings' width and density and good quality of the sawn timber. The test material was made from pure sapwood and heartwood and were thermally modified using the ThermoWood® method at VTT (Finland). A detailed description of the thermal modification method is reported in another publication (Metsä-Kortelainen et al., 2006). Thermal modifications were carried out at four different temperatures (170°C, 190°C, 210°C and 230°C) under a steam atmosphere. The time of thermal modification at the target temperature was 3 h in every test run. Specimens used in the decay tests were sawn out from 1.2 m long planks, but not until the thermal modifications were carried out. In addition, untreated wood material from four different wood species (Siberian larch, merbau, bangkirai and WRC) was used as reference material in the softrot test. These wood materials were industrial kiln dried and delivered by a Finnish timber company.

# Decay test against soft-rot fungi

The natural durability against the soft-rot microfungi of untreated and thermally modified sapwood and heartwood of Scots pine and Norway spruce and reference wood species (Siberian larch, merbau, bangkirai and WRC) was determined according to CEN/TS 15083-2 (2005). Small specimens ( $5 \times 10 \times 100 \text{ mm}^3$ ) were inserted into containers with unsterile soil. The incubation time was 32 weeks.

The test specimens of sapwood and heartwood of Scots pine and Norway spruce were sawn from eight different 1.2 m long test planks of each wood material thermally modified at VTT; 40 replicate specimens from each wood material were used, of which 30 were used in the decay test and 10 were used in determination of moisture content. In addition, 30 reference specimens of Scots pine sapwood were prepared. Specimens of other reference wood species were sawn from one to three replicate planks. The total number of test specimens was 720 and the number of moisture content specimens was 240.

All the specimens were conditioned at 65% relative humidity (RH) and 20°C to constant mass and weighed. The moisture content specimens and is the span (mm),  $w_2 - w_1$  is the increment of deformation corresponding to  $F_2 - F_1$  (mm), b is the width of the specimen (mm), and h is the height (thickness) of the specimen (mm).

The containers were incubated in a conditioning chamber at 27°C and 70% RH. After 32 weeks of incubation the specimens were cleaned of adhering soil particles and weighed. The MOE was determined in a similar way to before the incubation and the specimens were then dried at 103°C for 24 h and weighed. The moisture content after the test, and the mass and MOE loss were calculated and expressed as a percentage of their initial values.

The results of the soft-rot test were classified into durability classes based on the calculated X value (eq. 2). The durability classes are shown in Table I.

$$X = \frac{\text{Median value of MOE loss for test timber specimens}}{\text{Median value of MOE loss for reference timber specimens}}$$
 (2)

reference specimens were dried at 103°C for 24 h and weighed. The moisture content was calculated by expressing the mass of water  $(w_{\rm u}-w_{\rm dry})$  as a percentage of the oven-dry mass  $(w_{dry})$ . The mean moisture content of the moisture content specimens was calculated and used in the calculation of the initial dry mass of each test timber specimen according to CEN/TS 15083-2 (2005).

The test timber specimens and the reference timber specimens were impregnated with water and left in the vessels for 2 h. The modulus of elasticity (MOE) was determined with a static bending test using a central loading method according to EN 408 (1995). The bending test was carried out very carefully without breaking the specimens using a span of 80 mm, a loading speed of 1 mm min<sup>-1</sup> and maximal deformation of 0.5-1 mm, and the slope was calculated between the flexure load difference of 10-40 N on the straight line portion of the deformation curve. The MOE was calculated according to eq. (1). After the bending test the specimens were conditioned to approximately 50% moisture content based on their initial dry mass and dipped 80 mm vertically in containers with unsterile soil. The soil was compost based and its water-holding capacity was 51.7%.

MOE (Nmm<sup>-2</sup>) = 
$$\frac{(F_2 - F_1) \times l^3}{4 \times (w_2 - w_1) \times b \times h^3}$$
 (1)

where  $F_2 - F_1$  is the increment of load on the straight-line portion of the deformation curve (N), l

Table I. Durability rating scale according to CEN/TS 15083-2

Durability class	Description	X value
1	Very durable	≤0.10
2	Durable	$> 0.10$ to $\leq 0.20$
3	Moderately durable	$> 0.20$ to $\leq 0.45$
4	Slightly durable	$> 0.45$ to $\leq 0.80$
5	Not durable	>0.80

Decay test against brown-rot fungi

Thermally modified sapwood and heartwood of Scots pine and Norway spruce were exposed to brown-rot fungi C. puteana and Poria placenta essentially according to a mini-decay test (Bravery, 1979). The sample size was smaller and incubation time shorter than normally is used in the standardized tests [EN 350-1 (1994), EN 113 (1996) and CEN/TS 15083-1 (2005)]. In the previous studies on resistance of pine heartwood, similar tests with smaller specimens and shorter incubation times have been performed by VTT and published by Harju et al. (2003), Venäläinen et al. (2003) and Viitanen et al. (2006). A relevant dependence between mass loss and incubation time used in these studies has been detected.

The size of the test specimens was  $5 \times 20 \times$ 35 mm<sup>3</sup> and incubation times were 6 and 10 weeks. Four replicates for every wood material were used in the test with P. placenta (FPRL 280) and eight with C. puteana (BAM, Ebw 15). The total number of specimens was 640. The specimens were conditioned at 65% RH and 20°C to constant mass and then dried at 60°C for 48 h, after which they were cooled in a desiccator and weighed to an accuracy of 1 mg. The specimens were sterilized under steam as mentioned in the standard EN 113 (1996) and CEN/TS 15083-1 (2005), and subjected to a minidecay test on agar using rubber underlay between the agar and wood sample. After the decay test the specimens were weighed and then dried at 103°C for 24 h and weighed again for the calculation of mass loss and moisture content according to CEN/TS 15083-1 (2005).

The results of the brown-rot test were classified into durability classes based on the median mass losses according to CEN/TS 15083-1 (2005). The durability classes are shown in Table II.

Table II. Durability rating scale according to CEN/TS 15083-1 (2005).

Durability class	Description	% loss in mass		
1	Very durable	≤5		
2	Durable	>5 to ≤10		
3	Moderately durable	>10 to ≤15		
4	Slightly durable	$> 15$ to $\le 30$		
5	Not durable	>30		

# Results and discussion

Soft-rot test

The density of the soft-rot test specimens (at 65% RH and  $20^{\circ}$ C, nominal dimensions), moisture content before and after the soft-rot test, median MOE loss, calculated X value and durability class of the soft-rot decay test samples are presented in Table III. Each value is the mean of 30 replicate specimens. The median MOE loss of the reference specimens of pine sapwood was 64.8% (this value was used in calculation of the X values) and the mean MOE loss of the same samples was 64.9%, which exceeds the minimum validity limit (40%) of the decay test according to the standard CEN/TS 15083-2 (2005). In addition, the presence of soft rot was confirmed under light microscopy.

The mass and MOE losses in the soft-rot test are presented in Figures 1–5. Each column or point represents the average value and standard deviation of 30 replicates. The correlation between the two different evaluation methods, mass loss and MOE loss, is shown in Figure 6.

Table III shows that the density of the spruce and pine was reduced as a consequence of the weight loss taking place during the thermal modification. In addition, the reduction in the equilibrium moisture content (65% RH and 20°C) strongly depends on the thermal modification temperature. The moisture content of the thermally modified samples after the test was slightly lower than that of the untreated ones. This dependence of equilibrium moisture content on the level of thermal modification was first shown by Viitaniemi and Jämsä (1996).

In general, the thermal modification increased the decay resistance of the wood material (Figures 1–4). The mass loss and MOE loss differences between the sapwood and the heartwood were more evident with pine than with spruce. The differences between sapwood and heartwood of spruce were not significant. It seems that a sufficiently high thermal modification temperature of 210–230°C reduces the differences between different wood materials and gives clearly better decay resistance.

According to the present results from the soft-rot test in soil contact, the untreated heartwood of pine is also classified in the lowest durability class 5, as are the untreated pine sapwood and both spruce materials (Table III). In general, only bangkirai and merbau were more durable than heartwood of pine (Figure 5). A very interesting observation is that the mass loss of larch was at the same level as the values of untreated pine sapwood and spruce. However, the MOE loss of larch was even better than that of untreated pine heartwood and therefore larch was classified in durability class 4 (Table III).

Thermal modification very significantly affects the biological durability. The durability classes 2 and 3 in the soft-rot test can be reached with both spruce materials and sapwood of pine, respectively, by means of thermal modification at high enough temperatures, over 230°C. Pine heartwood thermally modified at this very high temperature can be classified into the best durability class 1. Thermal modification at the lower temperatures of 190°C and 210°C, which are commonly used in Finnish industry, increases the biological durability of pine sapwood and spruce (Figures 1–3), but the wood material is still classified into the durability classes "not durable" or "slightly durable" (Table III).

According to the standard EN 350, parts 1 and 2 (1994), untreated pine, spruce, larch, merbau, WRC and bangkirai are classified into the natural durability classes 3–4, 4, 3–4, 1–2, 2–3 and 2, respectively. The results of the soft-rot test seem to give worse durability classes than is determined for these wood species in the standard EN 350. The soft-rot test seems to be a very hard test because it was originally intended to measure the resistance of wood preservatives against soft-rot fungi. The test may even be too hard for wood species from durability class 3 or lower (4 and 5) to measure the natural durability.

Table III. Density, moisture content before and after decay test, median MOE loss, calculated X value and durability class of the soft-rot samples.

			Moisture co	ontent (%)			
	Thermal modification (°C)	Density (kg/m³)	RH 65%	After test	Median MOE loss (%) Test timber	X value	Durability class
Spruce, sapwood	Untreated	445.9	11.5	273.9	72.0	1.11	5
	170°C	430.5	9.8	266.0	71.4	1.10	5
	190°C	423.4	9.1	247.9	59.5	0.92	5
	210°C	412.6	7.4	231.3	36.9	0.57	4
	230°C	384.2	6.7	221.8	7.7	0.12	2
Spruce, heartwood	Untreated	434.3	11.6	276.4	69.8	1.08	5
	170°C	448.9	8.8	241.1	63.8	0.98	5
	190°C	429.1	9.0	235.3	52.8	0.81	5
	210°C	414.1	7.4	224.8	34.3	0.53	4
	230°C	381.4	5.8	221.1	7.3	0.11	2
Pine, sapwood	Untreated	501.3	11.4	247.0	69.6	1.07	5
	170°C	489.8	9.9	238.7	69.0	1.06	5
	190°C	478.6	8.5	229.2	65.8	1.01	5
	210°C	488.6	7.4	192.5	39.0	0.60	4
	230°C	475.8	6.3	181.9	19.9	0.31	3
Pine, heartwood	Untreated	554.1	11.0	192.4	53.6	0.83	5
	170°C	551.4	8.1	183.9	55.7	0.86	5
	190°C	524.4	7.9	182.3	43.5	0.67	4
	210°C	519.5	6.9	166.8	22.9	0.35	3
	230°C	466.7	5.7	175.3	6.6	0.10	1
Larch	Untreated	647.3	11.7	176.1	49.0	0.76	4
Bangkirai	Untreated	943.4	10.4	87.2	35.2	0.54	4
Merbau	Untreated	1119.1	10.3	61.2	21.0	0.32	3
WRC	Untreated	379.2	8.8	300.6	53.6	0.83	5

Note: MOE = modulus of elasticity; RH = relative humidity; WRC = western red cedar.

According to this research, differences between mass and MOE losses were, however, detected with untreated and thermally modified wood materials. Despite this, there were not equally important differences between durability classes of these wood materials rated with the scale determined in the standard, except for the results for thermally modified heartwood of pine.

The correlation between loss of mass and MOE is very high (Figure 6). This confirms the view that it is

Brown-rot test

The mass losses in the brown-rot tests with C. puteana and P. placenta are presented in Figures 7–10. Each value is the mean of four (P. placenta) or eight

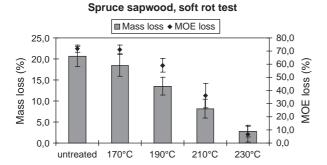


Figure 1. Mass and modulus of elasticity (MOE) losses of spruce sapwood after 32 weeks' incubation, soft-rot test.



(C. puteana) replicates. The median mass losses and

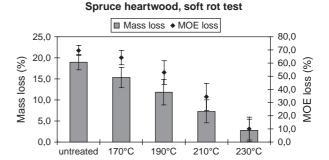


Figure 2. Mass and modulus of elasticity (MOE) losses of spruce heartwood after 32 weeks' incubation, soft-rot test.

possible to measure MOE loss many times during the incubation of the soft-rot test to measure the durability and resistance of the test samples.

#### Pine sapwood, soft rot test ■ Mass loss ◆ MOE loss 25,0 80.0 70.0 20,0 Mass loss (%) 60,0 8 50,0 loss 15,0 40,0 10,0 MOE 30.0 20,0 5,0 10,0 0,0 0.0 210°C untreated 170°C 190°C 230°C

Figure 3. Mass and modulus of elasticity (MOE) losses of pine sapwood after 32 weeks' incubation, soft-rot test.

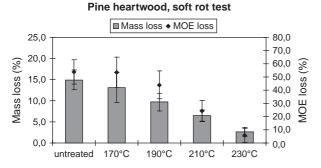


Figure 4. Mass and modulus of elasticity (MOE) losses of pine heartwood after 32 weeks' incubation, soft-rot test.

the durability classes according to CEN/TS 15083-1 (2005) are presented in Table IV.

In general, the heartwood of pine was also the most durable wood material in the brown-rot test. There was no significant difference between the decay resistance of sapwood and heartwood of spruce and sapwood of untreated pine. Thermal modification clearly increased the decay resistance to brown-rot fungi in all pine and spruce materials. The higher the thermal modification temperature, the more the biological durability was increased. This confirm the previous results on the positive effect of heat treatment on the decay resistance against brown rot (Viitanen *et al.*, 1994).

# Comparison wood species, soft rot test

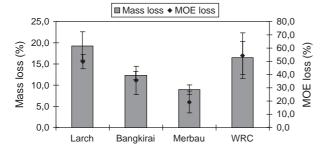


Figure 5. Mass and modulus of elasticity (MOE) losses of comparison wood species (Siberian larch, bangkirai, merbau and western red cedar) after 32 weeks' incubation, soft-rot test.

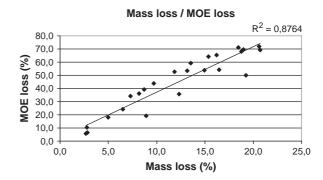


Figure 6. Correlation between mass loss and modulus of elasticity (MOE) loss, soft-rot test.

Poria placenta fungus was more aggressive than C. puteana against thermally modified wood, but after 10 weeks' exposure C. puteana caused mass losses in untreated wood the same as or higher than P. placenta. The differences between two brown-rot fungi were evident with spruce material when the thermal modification temperature was 210°C and the incubation time 10 weeks. The thermal modification of pine sapwood and heartwood at 210–230°C outstandingly improved the resistance to brown-rot fungi. The mass losses of all wood materials were near to 0 when the samples were modified at 230°C, and the decay resistance was comparable with the durability class "very durable" (Table IV).

The exposure time also significantly affected the mass losses and decay resistance (Figures 7–10). After 6 weeks' exposure the mass losses of different wood material were clearly smaller than after a longer incubation time. The exposure time may be used for the measurement of environmental severity during use of wooden products. In this study different exposure times were used for evaluating the biological resistance, and the results indicated that in the lower exposure situation of use class 3 (fences, façades) the durability of wood thermally modified at lower temperatures may be sufficient for acceptable requirements during its service life.

# General remarks on the results

On average, the biological durability of pine heartwood is better than that of pine sapwood and spruce, especially in use class 3 conditions, e.g. aboveground contact, but wide variation may exist (Viitanen *et al.*, 2006). In very severe exposure conditions, such as ground contact, the difference between the resistance of pine heartwood and that of pine sapwood and spruce is clearly smaller (Augusta & Rapp, 2003).

The intended use should be taken into account when determining the required biological durability.

# Spruce sapwood, brown rot test

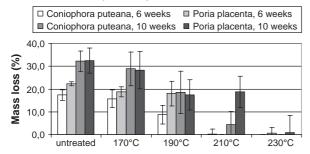


Figure 7. Mass loss of spruce sapwood after 6 and 10 weeks' brown-rot test with Coniophora puteana and Poria placenta.

## Spruce heartwood, brown rot test

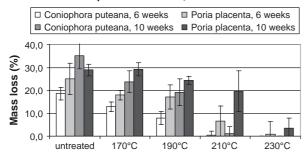


Figure 8. Mass loss of spruce heartwood after 6 and 10 weeks' brown-rot test with Coniophora puteana and Poria placenta.

# Pine sapwood, brown rot test

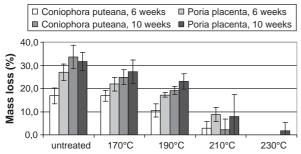


Figure 9. Mass loss of pine sapwood after 6 and 10 weeks' brownrot test with Coniophora puteana and Poria placenta.

# Pine heartwood, brown rot test

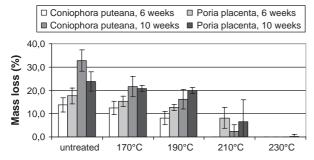


Figure 10. Mass loss of pine heartwood after 6 and 10 weeks' brown-rot test with Coniophora puteana and Poria placenta.

EN 335-1 (2006) presents the use classes representing different service situations to which wood and wood-based products can be exposed. It also indicates the biological agents relevant to each situation and use condition (1–5). The use condition in ground contact is ranked a high exposure condition (class 4) and the soft-rot test has been proposed to represent the biological exposure conditions under this use condition. The use class 3, wood above ground, is much wider and there are varied and very different use conditions. The test method using brown-rot fungi is proposed to represent partly this use condition, especially in the most exposed conditions such as terraces and jetties. However, in use class 3, the required resistance to decay is most often linked to the moisture damage conditions (decay damage) or high exposure situations. For these aspects, new opportunities to use the present test methods should be found and the results of different exposure times used during the tests should be taken into account more often for evaluating the durability needed in different intended use conditions. The coating may also have an important role in the resistance of wood against decay (Viitanen et al., 2006). The coating will decrease the water absorption and weathering effect, but in damage cases, the coating may also accelerate the decay if water is accumulated into the wood.

Thermal modification at very high temperatures (over 230°C) also has an effect on the other properties of wood. For instance, the strength properties are reduced, the wood becomes more brittle and the colour turns dark (Viitaniemi & Jämsä, 1996). Even though the biological durability of wood increases and water absorption decreases a great deal with thermal modification at high temperatures, the other properties may have an unfavourable effect on the usability of the wood material. The selection of the thermal modification temperature is a compromise between improved durability and reduced other properties. It can be concluded that thermal modification at 210°C does not reduce the strength properties significantly (Finnish ThermoWood Association, 2003), but it does improve the biological durability of Scots pine and Norway spruce to the level of merbau in the soft-rot test. Thermally modified pine heartwood in particular may be a potential material for applications where improved biological durability is needed.

# **Conclusions**

In general, both decay tests, soft-rot and brown-rot tests gave quite similar results. The untreated heartwood of Scots pine was more durable against decay organisms than the sapwood of Scots pine and the

Table IV. Median mass losses and the durability classes of the brown-rot test samples.

		Coniophora p	uteana, 6 weeks	Poria place	enta, 6 weeks	Coniophora pute	Coniophora puteana, 10 weeks		nta, 10 weeks
	Thermal modification (°C)	Median mass loss (%)	Durability class	Median mass loss (%)	Durability class	Median mass loss (%)	Durability class	Median mass loss (%)	Durability class
Spruce, sapwood	Untreated	17.4	4	22.0	4	31.3	5	32.6	5
	170°C	16.1	4	18.5	4	28.7	4	26.6	4
	190°C	9.6	2	18.0	4	20.8	4	14.9	3
	210°C	0.1	1	0.0	1	3.4	1	21.4	4
	230°C	0.0	1	0.0	1	0.0	1	4.6	1
Spruce, heartwood	Untreated	17.9	4	27.6	4	34.7	5	28.6	4
	170°C	13.3	3	17.2	4	24.2	4	28.3	4
	190°C	8.7	2	18.1	4	21.1	4	24.6	4
	210°C	0.0	1	7.5	2	0.2	1	22.1	4
	230°C	0.0	1	0.0	1	0.0	1	2.5	1
Pine, sapwood	Untreated	16.9	4	28.1	4	34.1	5	31.3	5
	170°C	15.7	4	22.3	4	24.8	4	27.6	4
	190°C	11.5	3	17.1	4	18.7	4	23.1	4
	210°C	2.1	1	9.0	2	0.3	1	8.1	2
	230°C	0.0	1	0.0	1	0.0	1	0.3	1
Pine, heartwood	Untreated	14.3	3	17.0	4	32.7	5	24.6	4
	170°C	13.9	3	16.3	4	21.9	4	21.2	4
	190°C	7.4	2	12.6	3	14.9	3	19.9	4
	210°C	0.0	1	8.8	2	1.9	1	3.5	1
	230°C	0.0	1	0.0	1	0.0	1	0.2	1

sapwood or heartwood of Norway spruce. The differences between sapwood and heartwood were more significant within pine than within spruce material. The thermal modification significantly increased the decay resistance of all pine and spruce samples. The higher the level of thermal modification, in other words the modification temperature, the better the biological durability. However, a very high thermal modification temperature, over 230°C, was needed to reach resistance equal to durability class 1 or 2 (durable or very durable) in the soft-rot test. Merbau was the most durable of the group of reference wood species and was classified into durability class 3 (moderately durable). The other reference wood species were classified into durability class 4 or 5 (slightly durable or not durable). The brown-rot test gave slightly better durability classes than the soft-rot test. Thermal modification at 210°C was enough to increase the durability of pine and spruce into durability classes 1 and 2 in almost every case.

The intended use should be taken into account in the evaluation of the test results and biological durability needed. One optional solution to evaluate the resistance level of different wooden products may be to use the test results after different exposure times during the accelerated tests.

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# **PUBLICATION IV**

# Effect of fungal exposure on the strength of thermally modified Norway spruce and Scots pine

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# **ORIGINAL ARTICLE**

# Effect of fungal exposure on the strength of thermally modified Norway spruce and Scots pine

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#### **Abstract**

Thermal modification at elevated temperatures changes the chemical, biological and physical properties of wood. In this study, the effects of the level of thermal modification and the decay exposure (natural durability against soft-rot microfungi) on the modulus of elasticity (MOE) and modulus of rupture (MOR) of the sapwood and heartwood of Scots pine and Norway spruce were investigated with a static bending test using a central loading method in accordance with EN 408 (1995). The results were compared with four reference wood species: Siberian larch, bangkirai, merbau and western red cedar. In general, both the thermal modification and the decay exposure decreased the strength properties. On average, the higher the thermal modification temperature, the more MOE and MOR decreased with unexposed samples and increased with decayed samples, compared with the unmodified reference samples. The strength of bangkirai was least reduced in the group of the reference wood species. On average, untreated wood material will be stronger than thermally modified wood material until wood is exposed to decaying fungi. Thermal modification at high temperatures over 210°C very effectively prevents wood from decay; however, strength properties are then affected by thermal modification itself.

**Keywords:** Bending strength, decay resistance, heartwood, modulus of elasticity, modulus of rupture, Norway spruce, sapwood, Scots pine, soft rot, thermal modification.

# Introduction

Thermal modification of wood is a process where the biological durability of wood is enhanced. In above-ground applications (use class 2 and 3 conditions), it is an alternative method for the traditional pressure impregnation of wood. Different methods for the thermal modification of wood have been developed in France, Finland, the Netherlands and Germany. One of these processes, developed in Finland, is referred to as the ThermoWood® process, which is environmental friendly with no toxic chemicals used. This method is based on heating the wood material for a few hours at high temperatures of over 180°C under normal pressure, using water vapour as a shielding gas.

Thermally modified wood is incorporated in many use class 2 and 3 applications, where enhanced dimensional stability and biological durability are needed, owing to high humidity exposure.

In addition, the brown colour of thermally modified wood is seen as a benefit in indoor furnishing. There are plenty of good experiences of using thermally modified timber in many different applications, such as exterior cladding, covered decking, flooring, garden furniture, panelling, kitchen furnishing, and the interiors of bathrooms and sauna baths. However, some problems have also been detected in existing applications with high moisture exposure, e.g. the wood material may have reached very high moisture content (MC), the surface of wood may have become unaesthetic, or the strength of the material may have been weakened and a plank may have suddenly broken. This confirms that more research is needed for the further development of thermal modification processes and to find out more detailed properties and suitable end-use applications of thermally modified wood.

The effect of thermal modification on the mechanical properties of wood has been widely studied.

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The change in mechanical properties is a consequence of changes in the wood's chemical composition. As a result of thermal modification, the wood becomes more brittle, and bending and tension strength decrease in relation to the level of thermal modification (Viitaniemi & Jämsä, 1996; Santos, 2000; Kamdem et al., 2002; Militz, 2002). In many studies, the bending strength [modulus of rupture (MOR)] was decreased significantly, whereas there was no or only a slight effect (decrease or increase) on the modulus of elasticity (MOE). For instance, Bekhta and Niemz (2003) heat-treated spruce wood at temperatures between 100 and 200°C, which decreased bending strength by 44-50%, while the modulus of elasticity was reduced by only 4–9%. In addition, Esteves et al. (2007a) found 40% and 50% decreases in bending strength for pine and eucalypt wood, respectively, while the MOE was little affected. Reduced strength properties have also been reported by Kubojima et al. (2000), Poncsák et al. (2006), Sundqvist et al. (2006), Boonstra et al. (2007a), Esteves et al. (2007b), Shi et al. (2007) and Korkut et al. (2008).

The improved fungal resistance of thermally modified wood has been reported by Viitanen et al. (1994), Sailer et al. (2000), Kamdem et al. (2002), Hakkou et al. (2006), Welzbacher and Rapp (2005, 2007), Jones et al. (2006), Mburu et al. (2006) and Boonstra et al. (2007b). However, depending on the level of the thermal modification, some degradation of wood components takes place in the event that thermally modified wood is exposed to fungal attack. Sivonen et al. (2003) studied the chemical properties of thermally modified Scots pine exposed to brownand soft-rot fungi and found that, as with the untreated wood, brown-rot fungi degraded mainly hemicelluloses while soft-rot fungus attacked cellulose more extensively. Mass loss caused by fungal attack was dependent on the modification temperature. Weiland and Guyonnet (2003) also found that in spite of strong hemicellulose degradation by the thermal modification, the fungal attack still takes place. In addition, the degradation of wood components caused by decaying fungi decreases the mechanical properties of wood (Fengel & Wegener, 1989; Curling et al., 2002).

In previous studies, the water absorption and the fungal resistance of sapwood and heartwood of Scots pine (*Pinus sylvestris*) and Norway spruce (*Picea abies*) thermally modified at four different temperatures were studied (Metsä-Kortelainen *et al.*, 2006; Metsä-Kortelainen & Viitanen, 2009). In general, thermal modification reduced the water absorption and increased the fungal resistance of wood in

relation to the level of the thermal modification. However, highly significant water absorption and mass loss differences between sapwood and heartwood of Scots pine and Norway spruce were detected in these studies. The differences between sapwood and heartwood of spruce were significantly smaller than with pine. According to these studies, it can be concluded that the effect of the wood part (sapwood/heartwood) has an important effect on the properties of wood, whether it is thermally modified or not.

It is known that both the thermal modification itself and the degradation of wood components in fungal exposure reduce the mechanical properties of wood. The strength of decayed thermally modified wood is a combination of these two parameters. The aim of this study was to determine the bending strength (MOR) and (MOE) of unexposed and softrotted sapwood and heartwood of Scots pine and Norway spruce thermally modified at several temperatures. The results of the bending test are compared with the results of water absorption test and decay test with soft-rot fungi using principal component analysis (PCA).

# Materials and methods

Materials

For the decay exposure in the laboratory and the bending strength test, pure sapwood and heartwood without juvenile wood of Norway spruce (Picea abies) and Scots pine (Pinus sylvestris) were selected from the Finnish sawmill industry. The target was to have research material with good quality and small variation in the width of year-rings. The selected wood material was industrially kiln-dried at an approximate temperature of 70°C to a MC of 11-15% before it was thermally modified at VTT using the ThermoWood® method. Thermal modifications were carried out at four different temperatures (170°C, 190°C, 210°C, 230°C) under a steam atmosphere. A detailed description of the selection of the research material and the thermal modification procedures is reported in another publication (Metsä-Kortelainen et al., 2006). In addition, industrially kiln-dried Siberian larch, merbau, bangkirai and western red cedar (WRC) were selected for the experiments as untreated reference material since these wood species are partly used in the same, mainly exterior, applications. The origin of this reference wood material is not known in more detail because it was delivered by a Finnish timber company.

Decay exposure and bending strength test

To determine the bending strength of the wood material, small specimens  $(5 \times 10 \times 100 \text{ mm}^3)$  were used. The longitudinal faces of the specimens were parallel to the direction of the grain. Half of the specimens chosen for the bending strength test were unexposed reference material conditioned at 65% relative humidity (RH) and 20°C to constant mass and weighed. The other half of the specimens for the strength test were selected from the material that had been exposed for 32 weeks to fungal attack in unsterile compost-based soil in accordance with CEN/TS 15083-2 (2005). This kind of soil and water content of the soil will expose wood mainly to soft-rotting fungi and bacteria. The presence of soft rot was confirmed under light microscopy after the exposure. Special visual signs of brown rot and white rot were not found in the studied samples or in the decay chambers. However, the eventual effects of these rot types are not critical for the evaluation of the results, since the potential effects of all types of decay on the strength properties of samples after different treatments are taken into consideration. After the natural durability test against the soft-rotting microfungi, these decayed specimens were also conditioned at 65% RH and 20°C to constant mass and weighed. Ten replicate specimens were taken from each wood material and the total number of test specimens was approximately 240. The original results of the decay test are reported in another publication (Metsä-Kortelainen & Viitanen, 2009).

The MOE and bending strength (MOR) were determined with a static bending test using a central loading method in accordance with EN 408 (1995). The bending test was carried out using a span of 80 mm and a loading speed of 1 mm min<sup>-1</sup>. The bending stress was directed to the middle of the upper face of the sample in a tangential direction. The slope of the decayed and reference material was calculated, in almost every case, between the flexure load differences of 10-40 N and 40-80 N on the straight-line portion of the deformation curve, respectively. Some of the specimens were very fragile and were less durable than the upper limit, and in these cases the slope was calculated manually. The MOE and MOR were calculated according to eqs (1) and (2):

MOE (N mm<sup>-2</sup>) = 
$$\frac{(F_2 - F_1) \times l^3}{4 \times (w_2 - w_1) \times b \times h^3}$$
 (1)

$$MOR (N mm^{-2}) = \frac{3 \times F \times l}{2 \times b \times h^2}$$
 (2)

where  $F_2 - F_1$  is the increment of load on the straight-line portion of the deformation curve (N), l is the span (mm),  $w_2 - w_1$  is the increment of deformation corresponding to  $F_2 - F_1$  (mm), b is the width of the specimen (mm), h is the height (thickness) of the specimen (mm), and F is the peak load (N).

After the bending strength test, the specimens were dried at 103°C for 24 h and weighed. The MC was calculated by expressing the mass of water  $(w_u - w_{dry})$  as a percentage of the oven-dry mass  $(w_{\rm dry})$ .

# Principal component analysis

The effects of wood properties [wood species, sapwood/heartwood, density, equilibrium moisture content (EMC), at 65% RH] and thermal modification (modification temperature, weight loss) on the durability (weight and MOE loss), strength (MOE, MOR) and water absorption (MC after 71 h floating) were analysed using PCA. PCA were carried out by Simca software. PCA is a multivariate projection method that is very useful for obtaining an overview of the dominant patterns and major trends in a large data group.

# Results and discussion

Initial data

The initial data of the test material are shown in Table I. The density (at 65% RH and 20°C, nominal dimensions) and weight loss caused by the thermal modification are presented therein. These values are average values of all specimens (reference and decay test specimens) before the decay test. In the same table, the EMCs at 65% RH of reference and decayed specimens, the results of water absorption test after 71 h floating and the results of the decay test against soft-rotting microfungi (weight and MOE loss) are also presented. These results are compared with the findings of the strength test presented later, using PCA.

In brief, Table I shows that the density of all spruce and pine samples was decreased as a consequence of reduced EMC and weight loss taking place during the thermal modification. Reduction in the EMC at 65% RH and 20°C after the thermal modification can be seen very clearly. However, the difference between the MCs of reference and decayed specimens is not very significant. More detailed information on the water absorption and decay test is presented in other publications

Table I. Initial data of the test material.

		Density (kg m <sup>-3</sup> )	Weight loss (%) After thermal modification	MC (%) RH 65%	MC (%) RH 65%	MC (%) After 71 h floating	Mass loss (%) After soft-rot test	MOE loss (%) After soft-rot test
	Thermal modification (°C)	Average	Average	Reference	Decayed	Reference	Decayed	Decayed
Spruce, sapwood	Untreated	444.6	0.0	11.0	10.2	19.0	20.2	72.6
	170°C	431.0	1.7	9.8	9.8	14.9	19.8	72.5
	190°C	426.5	3.5	8.9	8.9	14.0	13.4	58.7
	210°C	410.5	6.5	7.8	8.4	12.2	8.3	35.4
	230°C	387.2	10.1	6.2	7.1	10.1	2.9	5.6
Spruce, heartwood	Untreated	432.1	0.0	11.1	10.3	17.0	18.8	69.5
	170°C	441.8	3.4	8.6	9.7	12.8	16.3	64.6
	190°C	432.9	5.2	8.7	8.6	12.0	12.2	52.3
	210°C	421.9	6.7	6.9	8.3	9.8	8.2	36.3
	230°C	379.6	11.0	6.6	6.9	7.9	3.6	10.4
Pine, sapwood	Untreated	506.3	0.0	10.9	10.1	30.9	20.3	68.2
	170°C	493.1	2.0	9.2	9.7	42.5	18.3	66.3
	190°C	482.7	4.0	8.3	8.8	43.2	16.4	66.5
	210°C	490.2	6.6	7.2	8.4	37.2	8.8	38.0
	230°C	485.4	11.8	6.3	7.4	27.5	4.6	17.3
Pine, heartwood	Untreated	550.2	0.0	10.3	9.8	14.8	15.4	56.5
	170°C	540.2	5.2	8.2	8.8	9.6	13.9	55.6
	190°C	516.3	6.1	7.7	8.5	10.0	9.8	44.2
	210°C	512.3	7.1	7.0	7.8	10.2	6.4	23.9
	230°C	463.1	10.8	6.0	6.5	7.8	2.3	5.5
Larch	Untreated	634.3	_	10.3	10.1	_	19.3	49.9
Bangkirai	Untreated	915.3	_	8.7	8.9		11.6	29.9
Merbau	Untreated	1107.3	_	7.9	8.1		8.2	18.1
WRC	Untreated	372.3	-	7.8	9.5	-	16.6	50.6

Note: MC = moisture content; MOE = modulus of elasticity; RH = relative humidity; WRC = western red cedar.

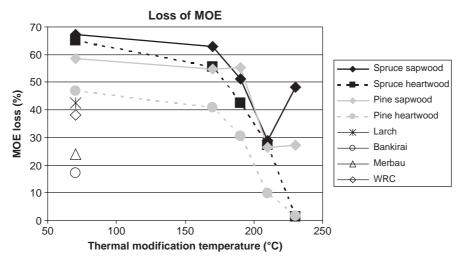


Figure 1. Modulus of elasticity (MOE) loss after fungal exposure. WRC = western red cedar.

(Metsä-Kortelainen et al., 2006; Metsä-Kortelainen & Viitanen, 2009).

# Strength test

The effect of fungal exposure on the MOE and MOR of the research material in a static bending test is presented in Figures 1 and 2. In general, both the MOE and MOR were reduced as a consequence of fungal exposure. The decrease in strength was greater with untreated wood (with the exception of the reference wood species) than with thermally modified samples. The higher the thermal modification temperature, the less the strength properties decreased as a consequence of fungal exposure.

The differences in MOE and MOR losses between sapwood and heartwood of spruce were quite small, except for sapwood samples thermally modified at 230°C (Figures 1 and 2). These samples had

exceptionally high MOE and MOR losses. This may partially be a consequence of contingency, because many of these samples suddenly broke at the early stages of the bending test. However, there is some congruence between both sapwood materials thermally modified at 230°C. Also, the strength loss of sapwood of pine modified at this very high temperature was only slightly changed compared with the results of samples modified at 210°C. On average, the losses of MOE and MOR were smaller with pine than with spruce. In addition, the differences between sapwood and heartwood of pine were more evident than with spruce. The loss of MOE of heartwood samples thermally modified at 230°C was negligible, and the bending strength, MOR, was only slightly affected. The MOE and MOR were moderately reduced with reference wood species. The strength was reduced least with bangkirai and merbau, which were approximately at the same level

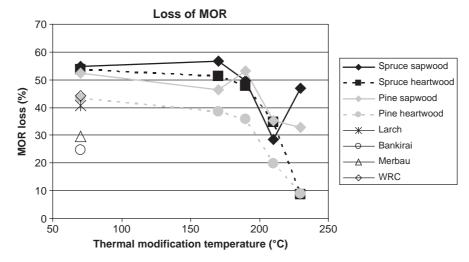


Figure 2. Modulus of rupture (MOR) loss after fungal exposure. WRC = western red cedar.

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Table II. Modulus of elasticity (MOE), standard deviation (SD) and effect of thermal modification (change) on unexposed and decayed wood material.

			Reference		Decayed			
	Thermal modification (°C)	MOE (N mm <sup>-2</sup> )	SD (N mm <sup>-2</sup> )	Change (%)	MOE (N mm <sup>-2</sup> )	SD (N mm <sup>-2</sup> )	Change (%)	
Spruce, sapwood	Untreated	11,294.8	1,056.4	0.0	3,708.9	507.5	0.0	
	170°C	10,104.3	1,150.4	-10.5	3,741.7	607.5	0.9	
	190°C	10,328.8	1,121.0	-8.6	5,030.2	485.0	35.6	
	210°C	9,573.4	762.0	-15.2	6,814.9	1,555.3	83.7	
	230°C	9,224.6	1,057.3	-18.3	4,779.4	2,160.0	28.9	
Spruce, heartwood	Untreated	10,647.8	910.3	0.0	3,723.4	554.8	0.0	
	170°C	10,901.7	1,578.6	2.4	4,836.3	904.2	29.9	
	190°C	10,607.2	1,151.5	-0.4	6,093.7	1,289.0	63.7	
	210°C	10,150.4	1,165.2	-4.7	7,380.2	1,175.2	98.2	
	230°C	8,156.3	1,530.8	-23.4	8,048.5	1,287.4	116.2	
Pine, sapwood	Untreated	10,587.2	1,299.3	0.0	4,378.9	603.8	0.0	
	170°C	10,729.7	1,498.1	1.3	4,858.6	1,109.3	11.0	
	190°C	10,790.1	1,025.0	1.9	4,833.4	565.0	10.4	
	210°C	10,888.1	943.4	2.8	8,007.4	1,150.2	82.9	
	230°C	10,699.3	1,076.9	1.1	7,775.0	3,248.0	77.6	
Pine, heartwood	Untreated	11,339.2	2,096.0	0.0	6,022.4	1,724.7	0.0	
	170°C	11,168.5	2,217.1	-1.5	6,599.2	2,282.4	9.6	
	190°C	10,977.0	1,333.0	-3.2	7,636.9	1,709.1	26.8	
	210°C	11,099.4	1,764.5	-2.1	10,023.5	1,849.1	66.4	
	230°C	10,086.6	1,369.6	-11.0	9,932.0	1,267.3	64.9	
Larch	Untreated	12,521.0	1,535.2	_	7,208.8	1,475.2	_	
Bangkirai	Untreated	15,896.0	1,695.0	_	13,154.9	3,451.0	-	
Merbau	Untreated	16,542.1	1,558.9	_	12,579.0	934.4	-	
WRC	Untreated	7,665.5	1,176.1	_	4,739.4	1,521.1	_	

Note: WRC = western red cedar.

as heartwood of spruce and pine thermally modified at the minimum at  $210^{\circ}$ C.

The average and standard deviation (SD) values of the MOE and MOR of the test material in the static bending test are presented in Tables II and III. The change values in the tables describe the effect of thermal modification on the strength properties. The strength values of thermally modified samples are compared with strength values of samples without thermal modification.

The MOE and the MOR of reference wood samples were decreased depending on the level of the thermal modification and wood species (Tables II and III). Both strength values were reduced more with spruce than with pine. The MOE and MOR of pine sapwood were only slightly affected (0-5%), depending on the modification temperature, and the changes in the strength values were very small with pine and spruce heartwood thermally modified at temperatures between 170 and 210°C. Thermal modification at 230°C decreased the MOE and MOR of spruce and pine heartwood by approximately 20% and 11-15%, respectively. The strength of spruce sapwood was most affected: thermal modification at 210°C reduced MOE and MOR 15%, and with samples thermally modified at 230°C

MOR was reduced nearly 30%. Bangkirai and merbau were the strongest reference wood species, while the MOE and MOR of WRC reached the lowest values of untreated wood in the whole study.

In general, MOE was reduced slightly less than MOR (Tables II and III). This is in agreement with Esteves et al. (2007a) and Bekhta and Niemz (2003). Viitaniemi and Jämsä (1996) also studied the effect of thermal modification on the bending strength of pine and spruce, and reported that MOR was lowered by 16% in spruce and by 12% in pine when the weight loss of wood material after thermal modification was approximately 11%. This is in quite good accordance with the results presented in this paper, where the weight losses of wood material after thermal modification at 230°C were approximately 11% (Table I). However, there may be some differences between the results of this paper and those of other publications, because the bending test was performed using smaller specimens than are usually used in strength tests.

Thermal modification increased the MOE and MOR of decayed wood material in almost every case (Tables II and III). The higher the modification temperature, the higher the MOE and MOR values. Thermal modification at least at 210°C increased the

Table III. Modulus of rupture (MOR), standard deviation (SD) and effect of thermal modification (change) on unexposed and decayed wood material.

			Reference		Decayed			
	Thermal modification (°C)	MOR (N mm <sup>-2</sup> )	SD (N mm <sup>-2</sup> )	Change (%)	MOR (N mm <sup>-2</sup> )	SD (N mm <sup>-2</sup> )	Change (%)	
Spruce, sapwood	Untreated	88.1	5.3	0.0	39.9	4.9	0.0	
	170°C	88.7	10.3	0.6	38.4	7.4	-3.8	
	190°C	85.7	16.8	-2.8	43.0	7.4	7.9	
	210°C	74.3	17.8	-15.7	53.2	17.2	33.3	
	230°C	64.2	18.1	-27.2	34.1	22.2	-14.6	
Spruce, heartwood	Untreated	87.3	4.8	0.0	40.4	6.0	0.0	
	170°C	93.1	10.6	6.6	45.3	9.9	12.1	
	190°C	95.5	9.7	9.4	50.0	10.0	23.9	
	210°C	86.3	19.1	-1.2	56.2	11.3	39.1	
	230°C	68.5	10.1	-21.5	62.6	11.6	55.1	
Pine, sapwood	Untreated	97.7	4.8	0.0	46.4	3.9	0.0	
	170°C	96.0	15.0	-1.7	51.4	10.1	10.9	
	190°C	94.7	13.2	-3.0	44.4	7.9	-4.3	
	210°C	93.7	18.0	-4.0	60.6	12.5	30.7	
	230°C	92.6	19.6	-5.1	62.3	29.4	34.2	
Pine, heartwood	Untreated	112.9	23.3	0.0	63.9	16.7	0.0	
	170°C	112.9	26.3	0.1	69.6	26.7	8.8	
	190°C	111.9	18.0	-0.8	72.0	17.7	12.6	
	210°C	109.1	18.9	-3.3	87.5	16.4	36.9	
	230°C	95.5	12.1	-15.3	87.1	14.5	36.2	
Larch	Untreated	135.3	24.1	_	80.2	18.2	_	
Bangkirai	Untreated	200.0	26.9	_	150.8	28.8	_	
Merbau	Untreated	234.9	33.8	_	165.5	14.5	_	
WRC	Untreated	79.0	13.6	_	44.0	14.7	_	

Note: WRC = western red cedar.

MOE by 65–116%, depending on the wood species, with the exception of spruce sapwood thermally modified at 230°C. The MOR of decayed wood material was increased by up to 55% as a consequence of thermal modification, although once again the sapwood of spruce underwent thermal modification at 230°C. There were also quite high SD values; in particular, the SD of spruce and pine sapwood thermally modified at 230°C was high in almost every case.

It can be seen from these results that both the thermal modification itself and fungal exposure affect the strength properties of wood. Untreated wood material will be stronger than thermally modified wood material until wood is exposed to decay fungi. Thermal modification at high temperatures over 210°C very effectively prevents wood from decaying, although the strength properties are affected by thermal modification itself. Edlund and Jermer (2004) studied the durability of heat-treated wood in the field according to EN 252 and observed that thermally modified stakes showed a high rate of failure after 2 years' exposure, but a microscopic analysis revealed no indication of decay. The authors concluded that the high rate of failure was a possible consequence of the strength loss caused by the

thermal modification, enhanced by wetting in the ground and further chemical degradation. In general, the same kind of behaviour can be detected from the results of this study (Table I). MOE loss was at a higher level than mass loss in the soft-rot test.

# Data analysis

The data on pine and spruce were classified using PCA. There were several variables in the PCA: the MOE, bending strength (MOR), density, weight loss caused by thermal modification, EMC, mass and MOE losses of the soft-rot test and the water absorption test results after floating for 71 h. The results of the data analysis are presented in score plots in which the information from loading plots is marked in text boxes. The score plot shows the sample distribution while the loading gives information about the distribution of the variables.

The data on spruce and pine were clustered differently (Figures 3 and 4). The data are ringed according to the thermal modification temperature. With spruce (Figure 3), the first principal component in the score plot sorted the samples out very clearly in accordance with their weight loss,

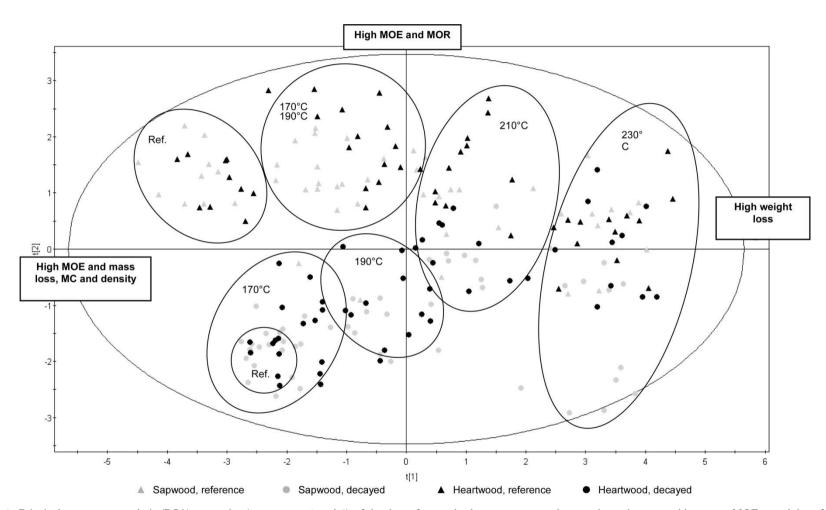


Figure 3. Principal component analysis (PCA) score plot (components 1 and 2) of the data of strength, decay exposure and water absorption tests with spruce. MOE = modulus of elasticity; MOR = modulus of rupture; MC = moisture content.

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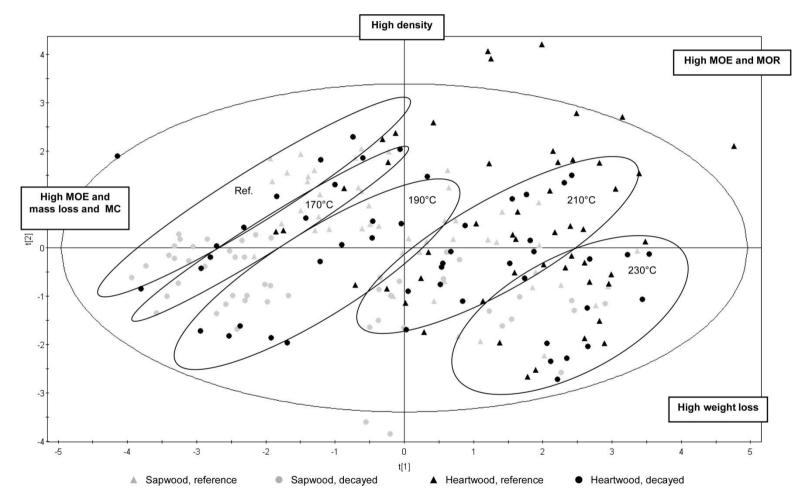


Figure 4. Principal component analysis (PCA) score plot (components 1 and 2) of the data of strength, decay exposure and water absorption tests with pine. MOE = modulus of elasticity; MOR = modulus of rupture; MC = moisture content.

durability, MCs and density. The samples with high weight loss in thermal modification are located on the right of Figure 3, while the samples with high mass and MOE loss values in the soft-rot test are located on the left. Samples with high moisture values (EMC and results of the floating test) are also located on the left. The strength values were clustered in accordance with the second principal component. The samples with high MOE and MOR are at the top of Figure 3. The sapwood and heartwood samples are dotted around the score plot, while the reference and decayed samples are located systematically: the decayed samples are at the bottom and the reference samples at the top of Figure 3.

The difference between sapwood and heartwood of pine was more distinguishable than for spruce (Figure 4). Most of the sapwood samples are located on the left of Figure 4, while the heartwood samples are on the right. The first principal component sorted the data according to the weight loss, strength, durability, MC and water absorption. The samples with high mass and MOE loss in the soft-rot test and high MCs are located on the left of Figure 4, while the samples with high weight loss in thermal modification and high MOE and MOR values are located on the right. The second principal component categorized the data in accordance with density. The samples with high density are at the top of Figure 4. The untreated and decayed samples of pine were not clustered as clearly as with spruce. The samples of both wood species were clustered in accordance with the thermal modification temperature. It is advantageous to note that the material properties and the effect of thermal modification of pine and spruce are different, not to mention every other wood species. A comprehensive understanding is needed to optimize the wood properties according to the circumstances and requirements of the application.

# Conclusions

The strength of the untreated and decayed thermally modified sapwood and heartwood of pine and spruce was examined. The results were compared with strength values of reference wood species (Siberian larch, merbau, bangkirai and WRC). Thermal modification decreased the MOE and the bending strength (MOR) of the unexposed reference samples depending on the level of thermal modification. The situation was reversed with decayed samples: the higher the thermal modification temperature was, the higher the MOE and MOR values were compared with values of unmodified samples. The strength differences between sapwood and heartwood of pine

were more evident than with spruce. Bangkirai and merbau were the strongest reference wood species.

On average, both the thermal modification itself and the fungal exposure reduced the strength of the wood. In general, the decrease in strength was approximately 0-30% and 0-65% as a consequence of thermal modification and fungal exposure, respectively. The fluctuation in strength loss was quite considerable as a consequence of the level of the thermal modification. However, the effect of decay exposure on the strength loss was more significant. Thus, it can be concluded that untreated wood material will be stronger than thermally modified wood material until wood is exposed to decaying fungi. Thermal modification at high temperatures of over 210°C quite effectively prevents wood from decay; however, the strength properties are then impacted to some extent by thermal modification itself. Other factors, e.g. high MC and defects in the wood material (knots, etc.), may also weaken the wood material in certain applications. The effect of moisture stress and wood defects may be more considerable in the case of thermally modified wood than with untreated wood. These impacts reflect the reasons why thermally modified wood is not recommended for use in load-bearing applications and why it should not replace pressure-treated

In many applications, adequate wood strength is required. The prevailing circumstances must be taken into account when choosing the wood material for a certain application. Structural wood protection has an important role, as do various kinds of surface treatments. Selection of the wood material in accordance with the demands of the application should always be based on knowledge and understanding of the material used.

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# **PUBLICATION VI**

# Durability of thermally modified Norway spruce and Scots pine in above ground conditions

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# **ORIGINAL ARTICLE**

# Durability of thermally modified Norway spruce and Scots pine in above-ground conditions

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#### Abstract

One of the main objectives of thermal modification is to increase the biological durability of wood. In this study the fungal resistance of Norway spruce and Scots pine, thermally modified at 195°C and 210°C, was studied with a lap-joint field test. Untreated pine and spruce and pine impregnated with tributyl tin oxide (TBTO) and copper, chromium and arsenic (CCA) were selected as reference materials. The evaluations were carried out after 1, 2 and 9 years of exposure. After 1 and 2 years of exposure mainly discoloration was detected. Only the untreated pine was slightly affected by decay fungi. There were significant differences in the decay ratings of untreated and thermally modified wood materials after 9 years in the field. While the untreated wood materials were severely attacked by decay fungi or reached failure rating, only small areas of incipient decay were detected in the thermally modified samples. Thermally modified pine was slightly more decayed than thermally modified spruce. The only wood material without any signs of decay was CCA-treated pine, since some of the TBTO-treated pine samples were also moderately attacked by fungal decay. The results of the lap-joint test had a good correlation with mass losses in a laboratory test with brown-rot fungi.

Keywords: Biological durability, brown rot, decay, discoloration, Norway spruce, Scots pine, thermal modification.

# Introduction

One of the main objectives of thermal modification is to increase the biological durability and dimensional stability of wood. Thermal modification changes the chemical composition of wood, thereby altering the appearance and the physical and biological properties of the wood. In the past two decades, the biological durability of thermally modified wood has been widely studied, mainly in laboratory conditions, and some methods based on elevated temperatures have been found to be quite effective in increasing the durability of wood against biological decay. For instance, Tjeerdsma et al. (2000) investigated the resistance of radiata pine, Scots pine, Douglas fir and spruce treated with the PLATO process at 160-190°C against two Basidiomycetes and soft-rot fungi and bacteria in a laboratory test and found a considerable improvement in durability compared with untreated wood materials. The degree of improvement varied between wood species. The PLATO process principally consists of two stages, hydrothermolysis and dry heat treatment, with an intermediate drying operation. There are also several publications about the improved durability of wood that has been thermally modified according to the principles of the retification process under a nitrogen atmosphere (Hakkou et al., 2006; Mburu et al., 2006, 2007; Lekounougou et al., 2009; Sušteršic et al., 2010) and with the oil-heat treatment (OHT) process in an oil bath (Sailer et al., 2000). Previous studies assessed the decay resistance of the sapwood and heartwood of Scots pine and Norway spruce that have been thermally modified at 170-230°C using the ThermoWood® method against soft and brown rot fungi, and found a good correlation between the thermal modification temperature and decay resistance (Metsä-Kortelainen & Viitanen, 2009). The ThermoWood® method is based on heating the wood material for a few hours at high temperatures above 180°C under normal pressure while protecting it with water vapour. The

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Table I. Decay rating.

Rating	Description	Definition
0	Sound	No softening or weakening of the wood
1	Moderate attack	Small areas of decay (softened, weakened wood); typically not more than 3 cm <sup>2</sup>
2	Severe attack	Marked softening and weakening of the wood typical of fungal decay; distinctly more than 3 cm <sup>2</sup> affected
3	Failure	Very severe and extensive rot; joint member(s) often capable of being easily broken

positive effects of different types of thermal modification on the biological resistance of several wood species in laboratory conditions are also reported in Viitanen et al. (1994), Viitaniemi (1997), Tjeerdsma et al. (1998), Kamdem et al. (2002), Gosselink et al. (2004), Boonstra et al. (2007) and Welzbacher et al. (2007). As concluded, the improvement in biological durability definitely depends on wood species, wood part (sapwood/heartwood), decay fungus, thermal modification method used and especially the intensity of thermal modification, which usually depends on the thermal modification temperature and time.

The biological durability of thermally modified wood has been assessed in many investigations using laboratory tests. However, the fungal resistance of thermally modified wood in ground contact and especially in above-ground contact field tests has been less studied. Welzbacher and Rapp (2005, 2007) investigated the biological durability of thermally modified wood industrially produced by different European suppliers with an EN 252 test in ground contact. They found that thermal modification improved natural durability in ground contact compared with untreated Scots pine sapwood; however, the rate of decay of the differently modified materials started to increase significantly after 3 years of exposure and the samples were attacked predominantly by white rot. Thermally modified samples were classified after 5.5 years of exposure only into the natural durability classes slightly durable (DC 4) or not durable (DC5), and the authors concluded that thermally modified wood appears to be unsuitable for ground contact applications. In the same research, the durability of thermally modified wood above ground was evaluated by means of a doublelayer test, in which some visible signs of decay were found only from one thermally modified material after 5.5 years of exposure (Welzbacher & Rapp, 2007). Edlund and Jermer (2004) also tested

thermally modified Scots pine and Norway spruce with an EN 252 and a ground proximity multiple layer test and after 2 years no visible signs of decay were detected. In the multiple-layer test, thermally modified wood seemed to be less susceptible to discolouring organisms than untreated wood.

Therefore, thermally modified wood is not recommended for use in ground contact. It is used extensively in applications without ground contact, such as in the decking and claddings of buildings. For this reason, in this study, the fungal resistance of thermally modified Norway spruce and Scots pine was investigated with a lap-joint test in the field. The wood material was thermally modified at temperatures corresponding to those used in the industrial production of thermally modified wood. The results were compared with those for untreated spruce and pine samples and impregnated wood materials.

# Materials and methods

For the horizontal lap-joint experiments carried out according to ENV 12037 (CEN, 1996), kiln-dried Norway spruce (Picea abies) and Scots pine (Pinus sylvestris) planks produced in south-eastern Finland were selected. The surfaces of the planks were sawn and their dimensions were  $50 \times 100 \,\mathrm{mm}^2$ . The selection criteria were small variation in the width of the year rings and good quality of the sawn timber and, in addition, that the planks had not been floated, water stored, or chemically treated or steamed. Part of the planks was selected for the thermal modifications, which were carried out under accurate conditions under steam at YTI Research Centre in Mikkeli, Finland. Two thermal modifications were carried out at 195°C and 210°C using the ThermoWood® method developed at VTT. The duration of thermal modification at the target temperature was 3h in both test runs. The

Table II. Discoloration rating.

Rating	Description	Definition
0	No discoloration	No evidence of discoloration caused by micro-organisms
1	Slight discoloration	Slight discoloration and/or some individual blue-stained spots
2	Distinct discoloration	Distinct discoloration, in groups of spots, streaks and/or patches of continuous staining
3	Total discoloration	Dark discoloration of entire surface area

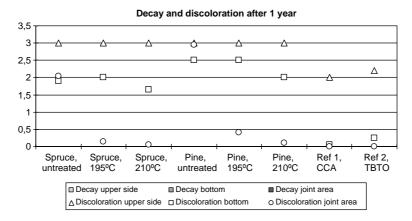


Figure 1. Decay and discoloration of the samples after 1 year of exposure. CCA = copper, chromium and arsenic; TBTO = tributyl tin oxide.

remainder of the spruce and pine planks was left untreated for use as reference material. In addition, Scots pine impregnated according to ENV 12037 (CEN, 1996) with tributyl tin oxide (TBTO) and copper, chromium and arsenic (CCA, class AB) was selected for the experiments.

Ten replicate specimens were prepared from each test material. The test covered eight different wood materials; therefore, the total number of test specimens was 80. The height of the specimens was 38 mm and the width 85 mm, and the length of specimen pairs was 300 mm with an overlapping close-fitting part at mid-length of 60 mm. The end grain surfaces of the specimens were sealed with weatherproof polyurethane sealant and the two joint members were fixed together with two cable straps. The specimens were then installed horizontally on purpose-designed exposure racks located approximately 1 m above ground level with the pith face downwards, at the test field of Otaniemi in southern Finland. The exposure started in November 2001 and the discoloration and decay of the specimens

were inspected separately after 1, 2 and 9 years, in accordance with the grading system presented in Tables I and II. The grading system was adapted from rating schemes in ENV 12037 (CEN, 1996) and EN 152 (CEN, 1984). Discoloration was evaluated visually and decay was inspected with a knife to reveal softened areas in the grading. In addition to investigating the differences between the selected materials, attention was paid to the suitability of the test method for modified wood, because the lap-joint method was originally developed for determining the relative protective effectiveness of wood preservatives.

# Results and discussion

Lap-joint specimens were inspected in November 2002, October 2003 and May 2010. The results for the development of discoloration and decay after different exposure times are presented in Figures 1– 3. The results for the upper sides, bottom sides and joint areas of the specimens are presented separately.

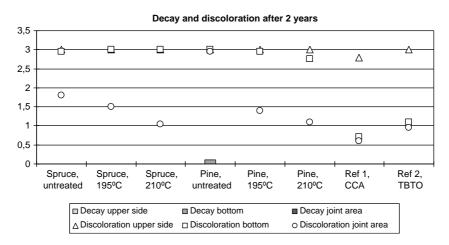


Figure 2. Decay and discoloration of the samples after 2 years of exposure. CCA = copper, chromium and arsenic; TBTO = tributyl tin oxide.

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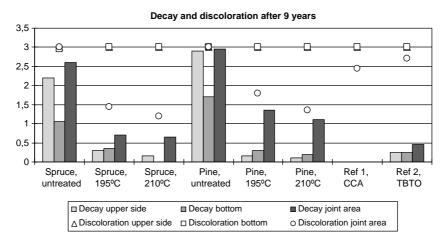


Figure 3. Decay and discoloration of the samples after 9 years of exposure. CCA = copper, chromium and arsenic; TBTO = tributyl tin oxide.

In general, the upper sides of the specimens were full of discoloration after 1 year in the field (Figure 1). Only the upper sides of the specimens treated with wood preservatives were not fully discolored. The bottom sides and joint areas were less discolored than the upper sides in all of the cases after the first evaluation. Thermal modification decreased the discoloration in the bottom sides and especially in joint areas. After 2 years of exposure, the difference between the discoloration of the upper sides and bottom sides was no longer significant, with the exception of impregnated samples (Figure 2). The joint area of untreated pine was also full of discoloration. All of the upper sides and bottom sides of the specimens were entirely discolored after 9 years in the field (Figure 3). In addition, the joint areas of the untreated spruce and pine were full of discoloration, as were the pressure-treated reference samples in practice. Thermal modification significantly decreased the discoloration of both wood species in the joint areas, which is in agreement with Edlund and Jermer (2004).

The first minor signs of decay were detected in untreated pine after 2 years of exposure in the field. Blom and Bergström (2006) ended up with the same type of results after testing Scots pine and Norway spruce in the field in above-ground conditions during 2 years: it seems that a longer period of exposure is needed to detect any decay damages in the wood material. After 9 years of exposure, highly significant differences in decay rating were discovered (Figure 3). In general, the joint areas of the specimens were mostly decayed. In addition, the upper sides and the bottom sides of the untreated spruce and pine samples were severely attacked by fungal decay. Both brown and white rot were detected. Most of these untreated wood samples and especially the pine samples reached failure rating and were full of decay and easily broken with gentle touching. As concluded, thermal modification improved substantially the fungal durability of both wood species. Thermally modified pine samples were somewhat more decayed than the comparable spruce samples. Thermal modification at 210°C

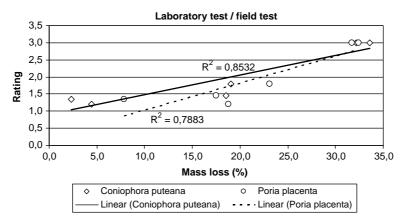


Figure 4. Correlation between mass loss in the laboratory test and rating of decay in joint areas in the field test. (Laboratory test data from Metsä-Kortelainen and Viitanen, 2009.)



Figure 5. Spruce samples (from the left: untreated, thermally modified at 210°C and 195°C) after 9 years of exposure. The upper and bottom sides of the specimens are at the bottom and top of the figure, respectively.

increased decay resistance slightly more than thermal modification at a lower temperature of 195°C. The only wood material without any signs of decay was CCA-treated pine, since some of the TBTO-treated pine samples were moderately attacked by fungal decay.

There has been discussion about the suitability of the present standard tests for evaluating the natural durability of wood and modified wood above ground and in ground contact. Råberg et al. (2005) concluded that accelerated tests should be used to



Figure 6. Pine samples (from the left: untreated, thermally modified at 210°C and 195°C) after 9 years of exposure. The upper and bottom sides of the specimens are at the bottom and top of the figure, respectively.



Figure 7. Pine samples [from the left: untreated, tributyl tin oxide (TBTO) and copper, chromium and arsenic (CCA) treated] after 9 years of exposure. The upper and bottom sides of the specimens are at the bottom and top of the figure, respectively.

complement long-term field tests and that laboratory tests are good as screening tests to obtain a first opinion about a new species or treatment. Thermally modified wood as well as acetylated or furfurylated wood should be regarded as a new wood species with improved natural durability (Jones et al., 2003). However, to determine the performance of modified wood, the standards and techniques currently used for assessing the preservative-treated wood are used, especially in the field. In our earlier study, the resistance of thermally modified Norway spruce and Scots pine against brown-rot fungi Coniophora puteana and Poria placenta were investigated with a mini-decay test (Bravery, 1979; Metsä-Kortelainen & Viitanen, 2009). The results were classified into durability classes according to CEN/TS 15083-1 (CEN, 2005). After 10 weeks of incubation, all of the sapwood samples of untreated spruce and pine were classified into the durability class not durable (DC 5), while the samples thermally modified at temperatures of 190°C and 210°C were classified in almost all cases into the classes moderately durable or slightly durable (DC 3-4) and very durable or durable (DC 1-2), respectively. The correlations between the mass losses after 10 weeks of incubation in the earlier laboratory test and the decay rating of the joint areas after 9 years of exposure in the lapjoint test of this study of untreated and thermally modified (at 190-195°C and 210°C) sapwood of spruce and pine are presented in Figure 4, where the samples thermally modified at 190°C and 195°C are regarded as having been treated at the same thermal modification temperature. The correlation is quite

high, especially with the results of the laboratory test with *C. puteana*. This indicates that a simple and quick laboratory test may give preliminary results concerning fungal durability in above-ground or use class 2–3 conditions. However, the laboratory tests only measure the decay damage in the wood, compared with field tests where the effect of the weather (rain, sunlight) on the performance of the wood can also be evaluated.

As a general discussion, it has to be mentioned that the discoloration of wood is partly a natural phenomenon as a consequence of ultraviolet light and it cannot be directly classified as a defect. However, in this test, the presence of blue stain was confirmed and green algae, lichen, Ditiola radicata and Dacrymyces stillatus fungi were also detected, particularly on the surfaces of the untreated and thermally modified samples. The bottom sides of the TBTO-treated samples were covered in mould. Most of the wooden samples were also full of cracks after 9 years of exposure; therefore, the strength and visual appearance of the samples decreased too (Figures 5-7). Both thermally modified and untreated wood rapidly turned grey as a result of exposure to sunlight. This is in agreement with Jämsä et al. (2000), who studied coated thermally modified wood in the field. Jämsä et al. (2000) concluded that the cracking of the thermally modified wood without a coating was at the same level as that of untreated wood despite the lower moisture content of the thermally modified wood. The weather resistance of thermally modified wood can be improved with commercial coatings.

In conclusion, the upper and bottom sides of both thermally modified wood and untreated wood discolored after a short exposure of 1–2 years in the field. Part of this discoloration was a consequence of blue stain, while part of it was due to the natural greying of wood. The bottom sides and joint areas of impregnated wood materials were not discolored until an exposure duration of 9 years. Thermal modification significantly decreased the discoloration of the joint areas, which were not exposed to direct sunlight. The samples that had been thermally modified at a higher temperature of 210°C were less discolored than samples modified at 195°C. Pine samples became discolored slightly more quickly than spruce samples.

The first signs of decay were detected in untreated pine after 2 years in the field, and after 9 years most of these samples reached failure rating. In addition, the untreated spruce samples were severely attacked by decay after 9 years of exposure. Thermal modification increased the biological durability of both wood species. In general, spruce was less attacked by decay than pine. The only wood material without

any signs of decay was CCA-treated pine, since some of the TBTO-treated pine samples were moderately attacked by fungal decay. The results of the lap-joint test correlated well with mass losses in a laboratory test with brown-rot fungi.

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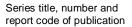
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Author(s)
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Title.

# Differences between sapwood and heartwood of thermally modified Norway spruce (*Picea abies*) and Scots pine (*Pinus sylvestris*) under water and decay exposure

Abstract

Thermal modification methods have been developed to increase the biological durability and dimensional stability of wood. The aim of this research was to study the differences between sapwood and heartwood of thermally modified Scots pine (*Pinus sylvestis*) and Norway spruce (*Picea abies*) under water and decay exposure. The effects of the modification temperature and wood coating were also examined.

Several tests were carried out in the laboratory and field. The main research material consisted of sapwood and heartwood of Scots pine and Norway spruce thermally modified at temperatures of 170°C, 190°C, 210°C and 230°C. The reference materials were untreated sapwood and heartwood of pine and spruce, larch, bangkirai, Western red cedar, merbau and pressure-treated wood materials, depending on the test.

Thermal modification decreased the water absorption of sapwood and heartwood of spruce in relation to the modification temperature in a floating test. The water absorption of sapwood and heartwood of pine either decreased or increased, however, depending on the modification temperature. Pine sapwood absorbed more water, and very quickly, than the other wood materials, whilst pine heartwood was the most water-repellent material in the test.

In general, the thermal modification increased the fungal durability in all the cases: the higher the modification temperature, the higher the resistance to fungal attack. Significant differences were detected between the different tests and wood materials. A very high thermal modification temperature (230°C) was needed to achieve resistance against decay comparable to that of the durability classes 'durable' or 'very durable' in the soft-rot test. The brown-rot test resulted in slightly better durability classes than the soft-rot test, which means that, already at lower temperatures (190–210°C), thermal modification clearly increases resistance to brown-rot attack, especially with pine materials. The results after nine years of exposure in the lap-joint field test had a good correlation with the results in the laboratory test with brown-rot fungi.

In this study, significant differences between the properties of thermally modified sapwood and heartwood of pine were detected in water and decay exposure. The differences between the sapwood and heartwood of spruce were notably smaller. The modification temperature had a remarkable effect on the properties of wood; this effect was not linear in every case however.

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Tekijä(t)

Sini Metsä-Kortelainen

Nimeke

# Lämpökäsitellyn kuusen (*Picea abies*) ja männyn (*Pinus sylvestris*) pinta- ja sydänpuun käyttäytyminen vesi- ja lahorasituksessa

Tiivistelmä

Puun lämpökäsittelymenetelmiä on kehitetty kosteuselämisen vähentämiseksi ja biologisen kestävyyden parantamiseksi. Tämän tutkimuksen tavoitteena oli selvittää lämpökäsitellyn männyn (*Pinus sylvestris*) ja kuusen (*Picea abies*) pinta- ja sydänpuun eroja kosteus- ja lahorasituksessa. Myös lämpökäsittelylämpötilan ja puun pintakäsittelyn vaikutusta tutkittiin.

Useita kokeita tehtiin sekä laboratoriossa että koekentällä. Useimmissa kokeissa käytetty tutkimusmateriaali koostui neljässä eri lämpötilassa (170 °C, 190 °C, 210 °C ja 230 °C) käsitellystä männyn ja kuusen pinta- ja sydänpuusta. Vertailumateriaalina oli kokeesta riippuen käsittelemätöntä männyn ja kuusen pinta- ja sydänpuuta, lehtikuusta, bangkiraita, jättiläistuijaa, merbauta sekä painekyllästettyjä puumateriaaleja.

Lämpökäsittely vähensi kuusen pinta- ja sydänpuun vedenimeytymistä käsittelylämpötilasta riippuen kellutuskokeessa. Männyn pinta- ja sydänpuulla puolestaan vedenimeytyminen kellutuskokeessa joko kasvoi tai väheni eri lämpötiloissa tehdyn käsittelyn seurauksena. Männyn pintapuu imi vettä runsaammin ja nopeammin kuin muut puumateriaalit, kun taas männyn sydänpuu imi itseensä kaikkein vähiten vettä.

Lämpökäsittely paransi yleisesti kaikkien puumateriaalien lahonkestoa. Mitä korkeampi oli käsittelylämpötila, sitä enemmän lahonkesto parani. Lahonkeston kasvussa oli kuitenkin merkittäviä eroja eri materiaalien välillä. Multalaatikkokokeessa tarvittiin lahonkestoluokkien "kestävä" tai "erittäin kestävä" saavuttamiseksi lämpökäsittely katkolahoa vastaan kaikkein korkeimmassa 230 °C:n lämpötilassa. Ruskolahokoe antoi yleisesti hieman parempia tuloksia, mikä tarkoittaa, että lämpökäsittely jo alemmissa (190–210°C) lämpötiloissa paransi etenkin männyn lahonkestoa merkittävästi. Kenttäkokeen tuloksilla yhdeksän vuoden rasituksen jälkeen oli hyvä korrelaatio laboratoriossa tehdyn ruskolahokokeen tulosten kanssa.

Tässä tutkimuksessa havaittiin merkittäviä eroja kosteus- ja lahorasituksessa lämpökäsitellyn männyn pinta- ja sydänpuun välillä. Erot kuusen pinta- ja sydänpuun välillä olivat huomattavasti pienemmät. Myös käsittelylämpötila vaikutti merkittävästi puun ominaisuuksiin, joskin on huomattava ettei lämpötilan vaikutus ollut lineaarista kaikissa tapauksissa

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Thermal modification methods have been developed to increase the biological durability and dimensional stability of wood. The aim of this research was to study the differences between sapwood and heartwood of thermally modified Scots pine (*Pinus sylvestis*) and Norway spruce (*Picea abies*) in water and decay exposure.

The main research material consisted of sapwood and heartwood of Scots pine and Norway spruce thermally modified at temperatures of 170°C, 190°C, 210°C and 230°C. The water absorption, contact angles, strength and decay resistance to several fungus types in the laboratory and field of the test material were studied.

The main conclusion derived from the results is that wood species, sapwood and heartwood portions and thermal modification temperature obviously have an influence on the biological and physical properties of thermally modified wood. These factors should be taken into account in the production processes and applications as well as in the testing.