Dissertation

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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 170ºC, 190ºC, 210ºC and 230ºC. 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 (230ºC) 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 (230ºC) 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 (190–210ºC), 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*) pinta- ja 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 rasi-

tuksen 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.

Contents

Publications I and V are not included in the PDF version. Please order the printed version to get the complete publication (http://www.vtt.fi/publications/index.jsp).

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 components 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 150ºC and 250ºC, 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 air-dried 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 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.

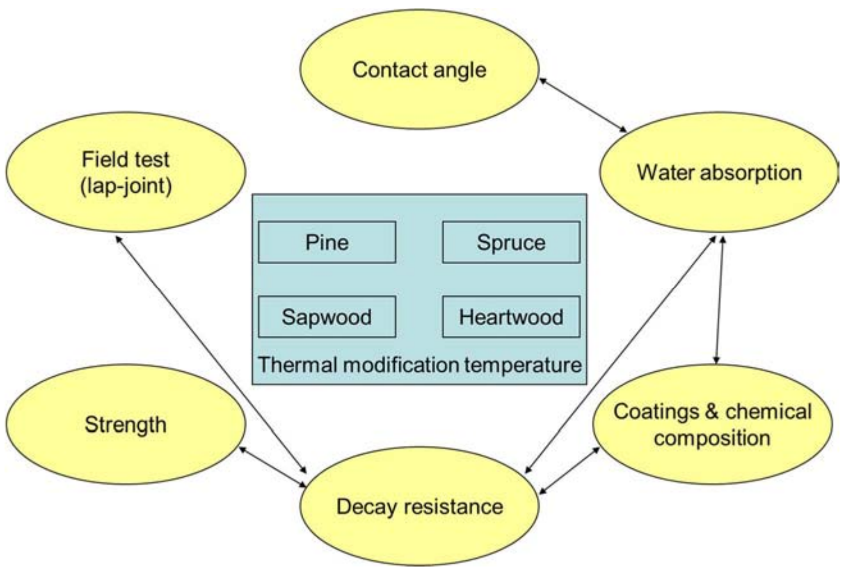

Figure 1. Schematic description of the wood materials of the main batch, the thermal modifications and the performed tests.

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.

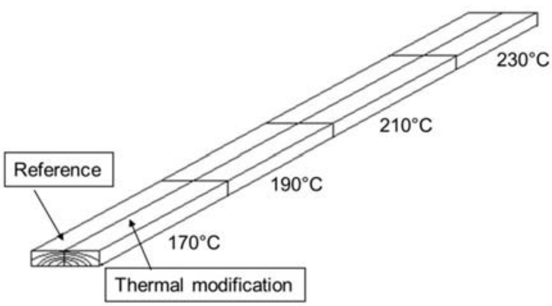

Figure 2. Cutting of the test planks for the thermal modifications.

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 60ºC. 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^2) 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: 170ºC, 190ºC, 210ºC and 230ºC, and 16 test runs were therefore carried out in total. Temperatures of 190ºC and 210ºC were selected to correspond to temperatures commonly used in industrial thermal modification processes (Thermo-S 190ºC, Thermo-

D 212ºC) and temperatures of 170ºC and 230ºC 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 195ºC and 210ºC.

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.

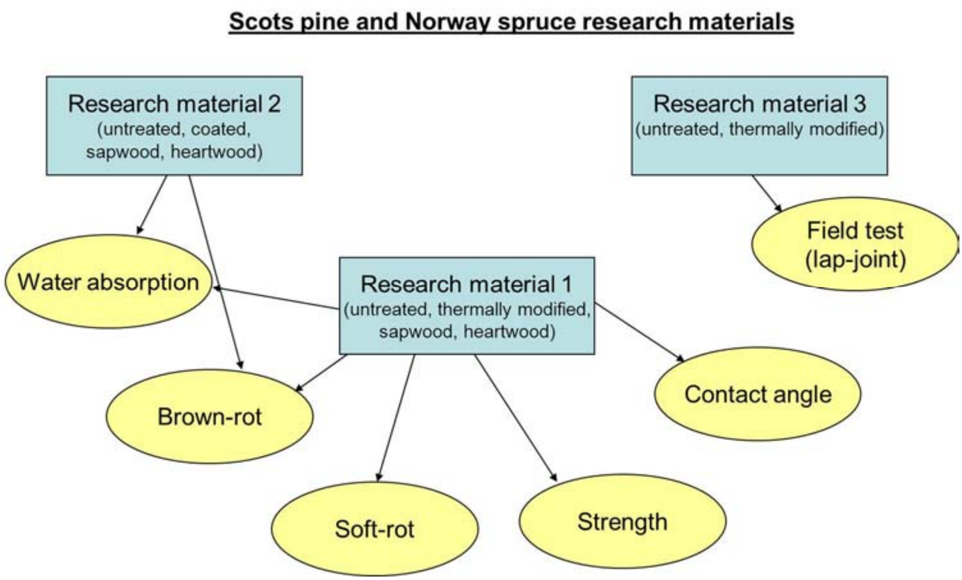

Figure 3. Research materials 1–3 and different tests performed in this study.

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 20ºC 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 x 20 x 35 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 mm^3) 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.

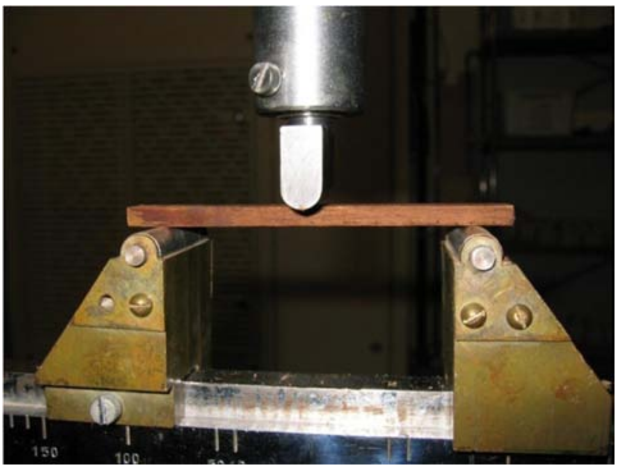

Figure 4. The static bending test, central loading method.

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 20ºC 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 (170ºC, 190ºC, 210ºC and 230ºC) 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 170–190ºC 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.

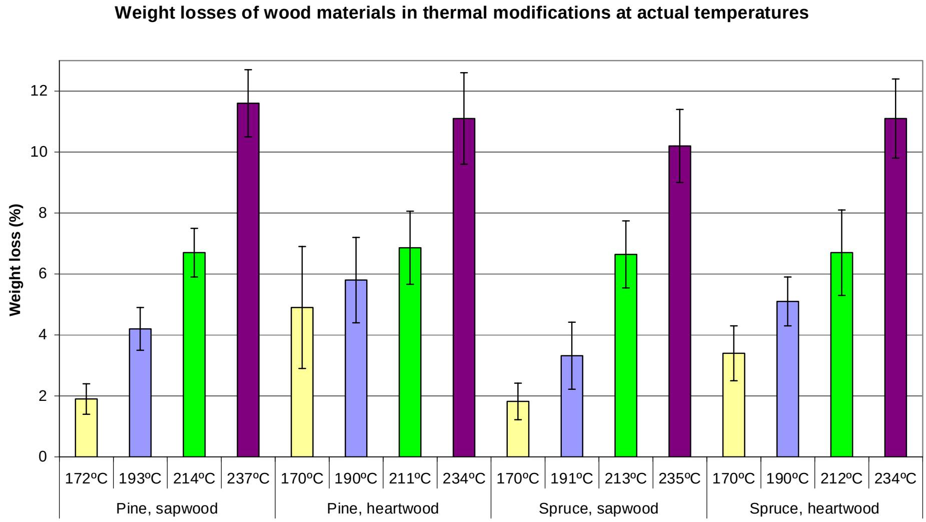

Figure 5. Weight losses and actual temperatures in thermal modification operations.

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 170–210ºC increased 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 180ºC and 220ºC for 2 and 4 hours and detected a similar increase in moisture content with samples thermally modified in milder conditions (180ºC, 2 hours). In another publication (Mohebby & Sanaei, 2005), the water absorption of thermally modified beech (*Fagus orientalis* Lipsky) at a temperature of 160ºC was decreased but increased after thermal modification at 180ºC 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.

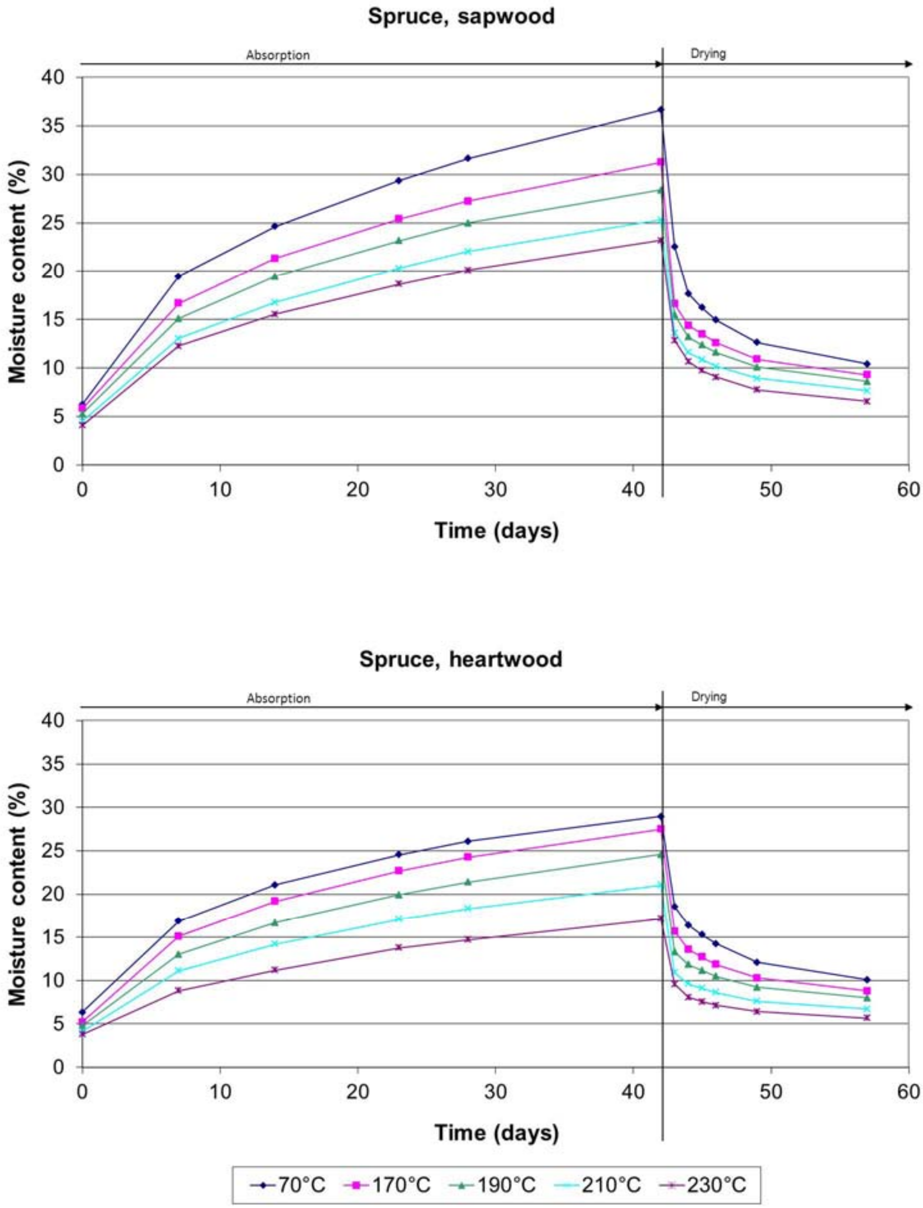

Figure 6. Extended water absorption of spruce sapwood and heartwood.

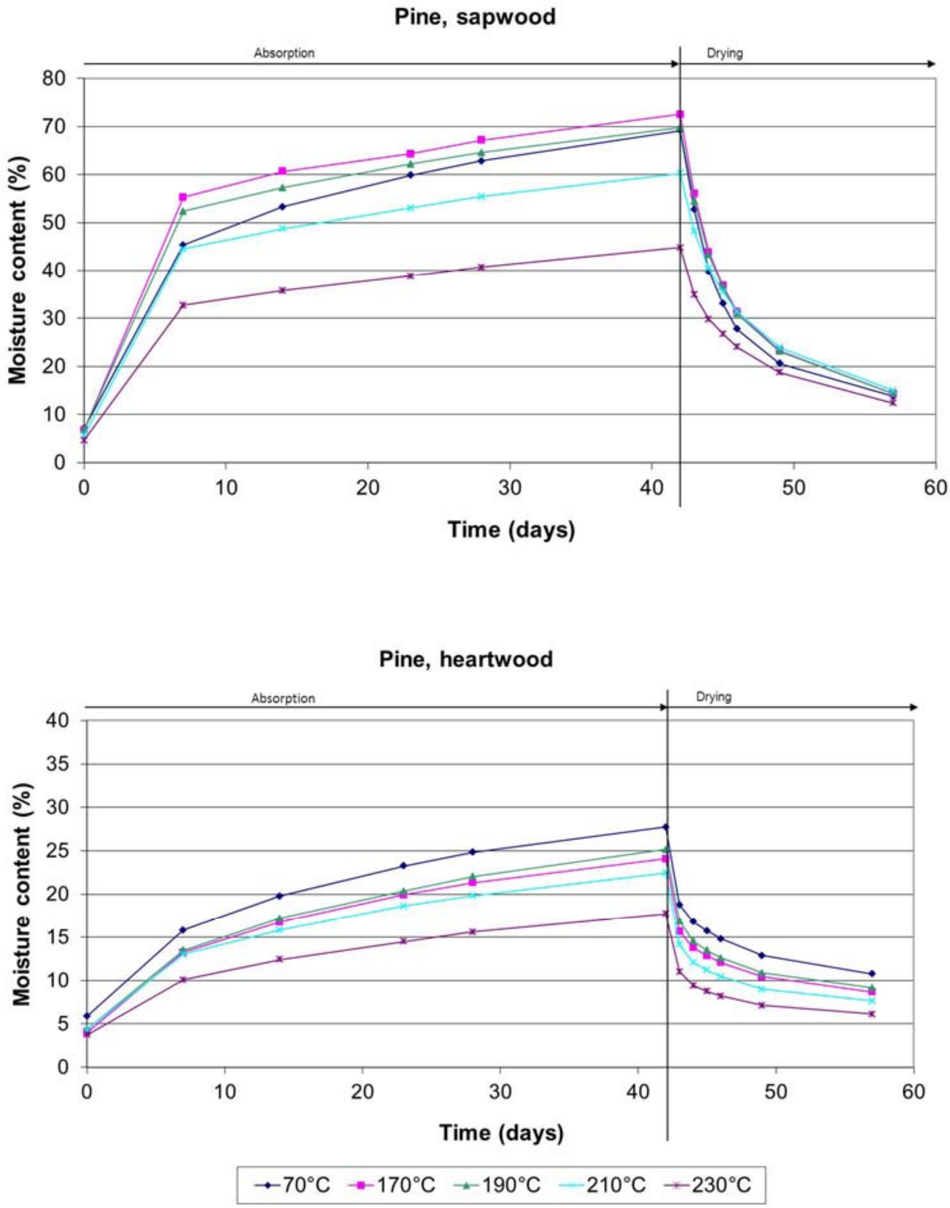

Figure 7. Extended water absorption of pine sapwood and heartwood.

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 170ºC, it then became more hydrophilic in the temperature range 190–210ºC until thermal modification at a very high temperature of 230ºC 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 230ºC were over 30%. Thermal modification at temperatures of 170–190ºC 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 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 200ºC. 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 230ºC 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 170ºC 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 170ºC. It is very interesting that thermal modification of pine at a temperature of 170ºC 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 230ºC 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 170ºC. 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.

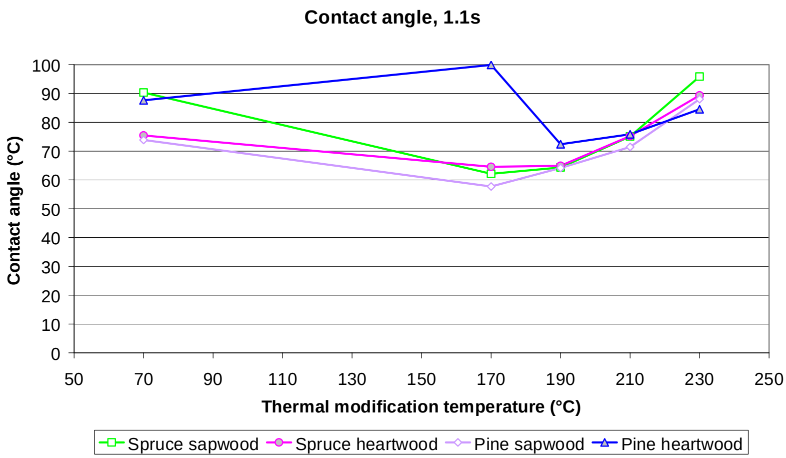

Figure 8. Contact angles of wood materials recorded after 1.1 seconds.

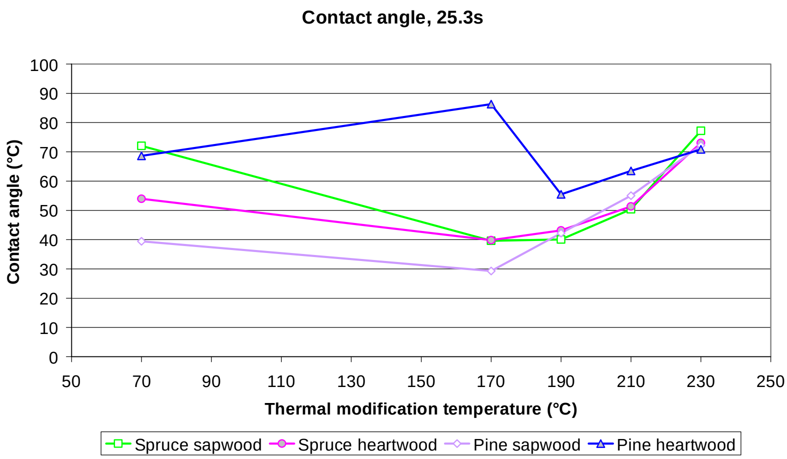

Figure 9. Contact angles of wood materials recorded after 25.3 seconds.

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.

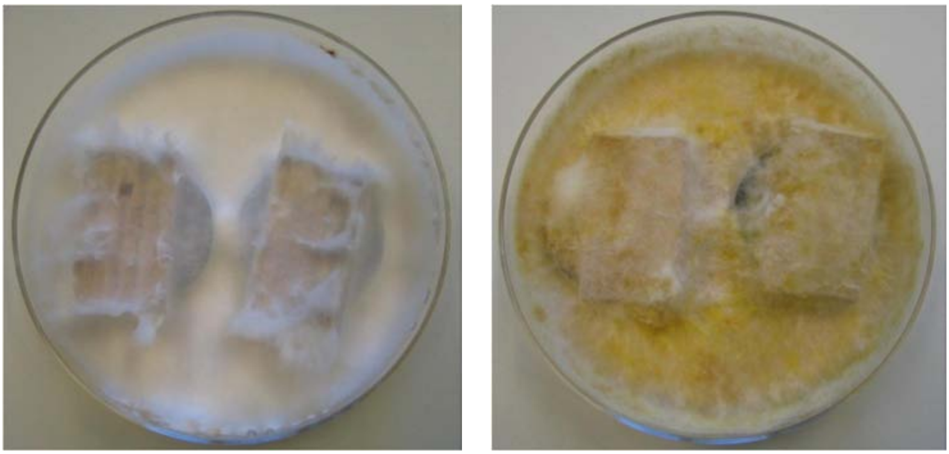

Figure 10. Brown-rot test samples, *Poria placenta* (left) and *Coniophora puteana* (right).

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 230ºC, the same materials were classified as 'very durable'. The thermal modification at temperatures of 190ºC and 210ºC, 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 (230ºC).

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 230ºC 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.

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 (%) RH65% Before test	MC (%) RH65% After test	MC (%) After test	MC (%) RH65% Before test	MC (%) RH65% After test	MC (%) 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 (%) RH65% Before test	MC (%) RH65% After test	MC (%) After test	MC (%) RH65% Before test	MC (%) RH65% After test	MC (%) 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 brown-rot 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 170–210ºC increases water absorption compared with unmodified wood.
- A relatively high thermal modification temperature (230ºC) 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.

References

Alén, R., Kotilainen, R. & Zaman, A. (2002). Thermochemical behavior of Norway spruce (*Picea abies*) at 180–225°C. Wood Science and Technology, 36, 163–171.

Almeida, G., Brito, J.O. & Perré, P. (2009). Changes in wood-water relationship due to heat treatment assessed on micro-samples of three Eucalyptus species. Holzforschung, 63, 80–88.

Andersson, S., Serimaa, R., Väänänen, T., Paakkari, T., Jämsä, S. & Viitaniemi, P. (2005). X-ray scattering studies of thermally modified Scots pine (*Pinus sylvestris L.*). Holzforschung, 59, 422–427.

Awoyemi, l. (2006). Heat treatment of less-valuable Nigerian-grown *Ceiba pentandra* wood for improved properties (Doc. No. IRG/WP 06-40332). International Research Group on Wood Preservation.

Bekhta, P. & Niemz, P. (2003). Effect of High Temperature on the Change in Color, Dimensional Stability and Mechanical Properties of Spruce Wood. Holzforschung, 57, 539–546.

Bergström, B., Gustafsson, G., Gref, R. & Ericsson, A. (1999). Seasonal changes of pinosylvin distribution in the sapwood/heartwood boundary of *Pinus sylvestris*. Trees, 14, 65–71.

Bergström, M. & Blom, Å. (2005). Above ground durability of Swedish softwood. Thesis for the degree of Doctor of Technology, Växjö University, Sweden 2005.

Boonstra, M.J. & Tjeerdsma, B. (2006). Chemical analysis of heat treated softwoods. Holz als Roh- und Werkstoff, 64, 204–211.

Boonstra, M.J., Van Acker, J. & Kegel, E. (2007a). Effect of a two-stage heat treatment process on the mechanical properties of full construction timber. *Wood Material Science and Engineering*, 3-4, 138–146.

Boonstra, M.J., Van Acker, J., Kegel, E. & Stevens M. (2007b). Optimisation of a two-stage heat treatment process: durability aspects. *Wood Science and Technology*, 41, 31–57.

Bravery, A.F. (1979). A miniaturised wood-block test for rapid evaluation of wood preservative fungicides (Doc. No. IRG/WP 2113). International Research Group on Wood Preservation.

Brischke, C., Welzbacher, C.R., Brandt, K. & Rapp, A.O. (2007). Quality control of thermally modified timber: Interrelationship between heat treatment intensities andCIE L*a*b* color data on homogenized wood samples. Holzforschung, 61, 19–22.

CEN/TS 15083-1 (2005). Durability of wood and wood-based products: Determination of the natural durability of solid wood against wood-destroying fungi, test methods. Part 1: Basidiomycetes. European Committee for Standardization.

CEN/TS 15083-2 (2005). Durability of wood and wood-based products: Determination of the natural durability of solid wood against wood-destroying fungi, test methods. Part 2: Soft-rotting micro-fungi. European Committee for Standardization.

Edlund, M.-L. & Jermer, J. (2004). Durability of heat-treated wood. COST action E22 Environmental Optimisation of Wood Protection, Lisboa – Portugal, 22nd–23rd March.

Elowson, T., Bergström, M. & Hämäläinen, M. (2003). Moisture dynamics in Norway spruce and Scots pine during outdoor exposure in relation to different surface treatments and handling conditions. Holzforschung, 57, 219–227.

EN 335-1 (2006). Durability of wood and wood-based products: Definition of use classes. Part 1: General. Brussels: European Committee for Standardization.

EN 350-1 (1994). Durability of wood and wood-based products: Natural durability of solid wood. Part 1: Guide to the principles of testing and classification of the natural durability of wood. Brussels: European Committee for Standardization.

EN 350-2 (1994). Durability of wood and wood-based products: Natural durability of solid wood. Part 2: Guide to natural durability and treatability of selected wood species of importance in Europe. Brussels: European Committee for Standardization.

EN 927-5 (2000). Paints and varnishes: Coating materials and coating systems for exterior wood. Part 5: Assessment of the liquid water permeability. Brussels: European Committee for Standardization.

ENV 12037 (1996). Wood Preservatives: Field test method for determining the relative protective effectiveness of a wood preservative exposed out of ground contact. Horizontal lap-joint method. Brussels: European Committee for Standardization.

Esteves, B., Domingos, I. & Pereira, H. (2007a). Improvement of technological quality of eucalypt wood by heat treatment in air at 170–200°C. *Forest Products Journal*, 57 (1/2), 47–52.

Esteves, B., Marques, A., Domingos, I. & Pereira, H. (2007b). Influence of steam heating on the properties of pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood. *Wood Science and Technology*, 41, 193–207.

Esteves, B., Graça, J. & Pereira, H. (2008a). Extractive composition and summative chemical analysis of thermally treated eucalypt wood. Holzforschung, 62, 344–351.

Esteves, B., Marques, A.V., Domingos, I. & Pereira, H. (2008b). Heat-induced colour changes of pine (*Pinus pinaster*) and eucalypt (*Eucalyptus globulus*) wood. Wood Science and Technology, 42, 369–384.

Esteves, B. & Pereira, H. (2009). Wood modification by heat treatment: A review. BioResources, 4, 370–404.

Fengel, D. & Wegener, G. (1984). Wood: Chemistry, Ultrastructure, Reactions. Berlin: Walter de Gruyter & Co.

Follrich, J., Müller, U. & Gindl, W. (2006). Effects of thermal modification on the adhesion between spruce wood (*Picea abies Karst.*) and a thermoplastic polymer. Holz als Roh- und Werkstoff, 64, 373–376.

Frühwald, E., Li, Y. & Wadsö, L. (2008). Image analysis study of mould susceptibility of spruce and larch wood dried or heat-treated at different temperatures. Wood Material Science and Engineering, 1–2, 55–61.

González-Peña, M.M. & Hale, M.D.C. (2009a). Colour in thermally modified wood of beech, Norway spruce and Scots pine. Part 1: Colour evolution and colour changes. Holzforschung, 63, 385–393.

González-Peña, M.M. & Hale, M.D.C. (2009b). Colour in thermally modified wood of beech, Norway spruce and Scots pine. Part 2: Property predictions from colour changes. Holzforschung, 63, 385–393.

González-Peña, M.M., Curling, S.F. & Hale, M.D.C. (2009). On the effect of heat on the chemical composition and dimensions of thermally modified wood. Polymer Degradation and Stability, 94, 2184–2193.

Gosselink, R.J.A., Krosse, A.M.A., van der Putten, J.C., van der Kolk, J.C., de Klerk-Engels, B. & van Dam, J.E.G. (2004). Wood preservation by low-temperature carbonisation. *Industrial Crops and Products*, 19, 3–12.

Gunduz, G., Aydemir, D. & Karakas, G. (2009). The effects of thermal treatment on the mechanical properties of wild Pear (*Pyrus elaeagnifolia Pall.*) wood and changes in physical properties. Materials and Design, 30, 4391–4395.

Hakkou, M., Pétrissans, M., El Bakali, I., Gérardin, P. & Zoulalian, A. (2005a). Wettability changes and mass loss during heat treatment of wood. Holzforschung, 59, 35–37.

Hakkou, M., Pétrissans, M., Zoulalian, A. & Gérardin, P. (2005b), Investigation of wood wettability changes during heat treatment on the basis of chemical analysis. Polymer Degradation and Stability, 89, 1–5.

Hakkou, M., Pétrissans, M., Gérardin, P. & Zoulalian A. (2006). Investigations of the reasons for fungal durability of heat-treated beech wood. Polymer Degradation and Stability, 91, 393–397.

Hale, M.D., Ghosh, S.C. & Spear, M.J. (2005). Effects of artificial UV weathering and soft rot decay on heat treated wood (Doc. No. IRG/WP 05-40302). International Research Group on Wood Preservation.

Harju, A.M. Venäläinen, M., Anttonen, S., Viitanen, H., Kainulainen, P., Saranpää, P. & Vapaavuori, E. (2003). Chemical factors affecting the brown-rot decay resistance of Scots pine heartwood. Trees, 17, 263–268.

Herajärvi, H. (2009). Effect of drying technology on aspen wood properties. Silva Fennica, 43(3), 433–445.

Homan, W., Tjeerdsma, B., Beckers, E. & Jorissen, A. (2000). Structural and other properties of modified wood. Proceedings of the World Conference on Timber Engineering, Whistler Resort, British Columbia, Canada, July 31st–August 3rd.

Homan, W.J. & Jorissen, A.J.M. (2004). Wood modification developments. Heron, 49, 361–386.

Humar, M., Bučar, B. & Pohleven, F. (2006). Correlation between modulus of elasticity, mass losses and FTIR spectra of copper treated decayed wood (Doc. No. IRG/WP 06-10580). International Research Group on Wood Preservation.

Jermer, J. & Andersson, B.-L. (2005). Corrosion of fasteners in heat-treated wood – progress report after two years' exposure (Doc. No. IRG/WP 05-40296). International Research Group on Wood Preservation.

Johansson, D. & Morén, T. (2006). The potential of colour measurement for strength prediction of thermally treated wood. Holz als Roh- und Werkstoff 64, 104–110.

Jämsä, S., Ahola, P. & Viitaniemi, P. (2000). Long-term natural weathering of coated ThermoWood. *Pigment & Resin Technology*, 29, 68–74.

Jämsä, S. & Viitaniemi, P. (2001). Heat treatment of wood: Better durability without chemicals. In: Review on heat treatments of wood, Proceedings of the special seminar of COST Action E22, Antibes, France.

Kamdem, D.P., Pizzi, A. & Triboulot, M.C. (2000). Heat-treated timber: potentially toxic byproducts presence and extent of wood cell wall degradation. Holz als Roh- und Werkstoff 58, 253–257.

Kamdem, D.P., Pizzi, A. & Jermannaud, A. (2002). Durability of heat-treated wood. Holz als Roh- und Werkstoff, 60, 1–6.

Kartal, S.N., Hwang, W.-J. & Imamura, Y. (2007). Water absorption of boron-treated and heat-modified wood. Journal of Wood Science, 53, 454–457.

Kocaefe, D., Shi, J.L., Yang, D.-Q. & Zhang, J. (2007). Preliminary study of thermal treatment effects on mold growth of selected Quebec wood species. Forest Products Journal, 57, 30–33.

Kocaefe, D., Poncsak, S., Doré, G. & Younsi, R. (2008). Effect of heat treatment on the wettability of white ash and soft maple by water. Holz als Roh- und Werkstoff, 66, 355–361.

Kollmann, F. & Côté, W. (1968). Principles of Wood Science and Technology: Solid Wood. Springer-Verlag, Berlin.

Korkut, S. & Bektaş, Î. (2008). The effects of heat treatment on physical properties of Uludaĝ fir (*Abies bornmuelleriana Mattf*.) and Scots pine (*Pinus sylvestris L*.) wood. Forest Products Journal, 58, 95–99.

Korkut, S., Akgül, M. & Dündar, T. (2008). The effects of heat treatment on some technological properties of Scots pine (*Pinus sylvestris L*.) wood. Bioresource Technology, 99, 1861–1868.

Kotilainen, R. (2000). Chemical changes in wood during heating at 150–260°C. Doctoral Thesis, University of Jyväskylä, Finland.

Kubojima, Y., Okano, T. & Ohta, M. (2000). Bending strength and toughness of heat-treated wood. *Journal of Wood Science*, 46, 8–15.

Kärkkäinen, M. (2003). Puutieteen perusteet (Principles of Wood Science). Karisto Oy, Hämeenlinna. (In Finnish.)

Lekounougou, S., Pétrissans, M., Jacquot, J.P., Gelhaye, E. & Gérardin, P. (2009). Effect of heat treatment on extracellular enzymatic activities involved in beech wood degradation by *Trametes versicolor*. Wood Science and Technology, 43, 331–341.

Majano-Majano, A., Hughes, M. & Fernandez-Cabo, J.L. (2010). The fracture touchness and properties of thermally modified beech and ash at different moisture contents. Wood Science and Technology, DOI 10.1007/s00226-010-0389-4.

Mburu, F., Dumarçay, S., Huber, F., Pétrissans, M. & Gérardin, P. (2006). Improvement of *Grevillea robusta* durability using heat treatment (Doc. No. IRG/WP 06-40333). International Research Group on Wood Preservation.

Mburu, F., Dumarçay, S., Huber, F., Pétrissans, M. & Gérardin, P. (2007). Evaluation of thermally modified *Grevillea robusta* heartwood as an alternative to shortage of wood resource in Kenya: Characterisation of physicochemical properties and improvement of bio-resistance. Bioresource Technology, 98, 3478–3486.

Mikleĉić, J., Jirouš-Rajković, V., Antonović, A. & Španić, N. (2011). Discolouration of thermally modified wood during simulated indoor sunlight exposure. BioResources, 6, 434–446.

Militz, H. & Tjeerdsma, B. (2001). Heat treatment of wood by the PLATO-process. In: Review on heat treatments of wood, Proceedings of the special seminar of COST Action E22, Antibes, France.

Mitchell, P. (1988). Irreversible property changes of small loblolly pine specimens heated in air, nitrogen or oxygen. Wood and Fiber Science, 20, 320–335.

Mohebby, B. & Sanaei, I. (2005). Influences of the hydro-thermal treatment on physical properties of beech wood (*Fagus orientalis*) (Doc. No. IRG/WP 05-40303). International Research Group on Wood Preservation.

Nuopponen, M., Vuorinen, T., Jämsä, S. & Viitaniemi, P. (2003). The effects of a heat treatment on the behaviour of extractives in softwood studied by FTIR spectroscopic methods. Wood Science and Technology, 37, 109–115.

Ohmae, Y., Saito, Y., Inoue, M. & Nakano, T. (2009). Mechanism of water adsorption capacity change of bamboo by heating. European Journal of Wood and Wood products, 67, 13–18.

Oliveira, R.M., Brisolari, A., Sales, A. & Gonçalves, D. (2010). Wettability, shrinkage and color changes of *Araucaria angustifolia* after heating treatment. Materials Research, 13, 351–354.

Olsson, T., Megnis, M., Varna, J. & Lindberg, H. (2001). Study of the transverse liquid flow paths in pine and spruce using scanning electron microscopy. Journal of Wood Science, 47, 282–288.

Petrič, M., Pavlič, M., Humar, M. & Tomažič, M. (2006). Blue staining of coated modified and unmodified wood (Doc. No. IRG/WP 06-10589). International Research Group on Wood Preservation.

Pétrissans, M., Gérardin, P., El bakali, I. & Serraj M. (2003). Wettability of Heat-Treated Wood. Holzforschung, 57, 301–307.

Phuong, L.X., Shida, S. & Saito, Y. (2007). Effects of heat treatment on brittleness of *Styrax tonkinensis* wood. Journal of Wood Science, 53, 181–186.

Poncsák, S., Kocaefe, D., Bouazara, M. & Pichette, A. (2006). Effect of high temperature treatment on the mechanical properties of birch (*Betula papyrifera*). Wood Science and Technology, 40, 647–663.

Popper, R., Niemz, P. & Eberle, G. (2005). Untersuchungen zum sorptions- und quellungsverhalten von thermisch behandelten holz. Holz als Roh- und Werkstoff, 63, 135–148.

Rapp, A.O. & Sailer, M. (2001). Oil heat treatment of wood in Germany: State of the art. In: Review on heat treatments of wood, Proceedings of the special seminar of COST Action E22, Antibes, France.

Repellin, V. & Guyonnet, R. (2005). Evaluation of heat-treated wood swelling by differential scanning calorimetry in relation to chemical composition. Holzforschung, 59, 28–34.

Saarelainen, U. (1981). Puurakenteet 1. Puu materiaalina. Rakentajain kustannus, Helsinki. (In Finnish.)

Sailer, M., Rapp, A.O., Leithoff, H. & Peek, R.-D. (2000). Vergütung von Holz durch Anwendung einer Öl-Hitzebehandlung. Holz als Roh- und Werkstoff 58, 15–22.

Santos, J.A. (2000). Mechanical behaviour of Eucalyptus wood modified by heat. *Wood Science and Technology*, 34, 39–43.

Schnabel, T., Zimmer, B., Petutschnigg, A.J. & Schönberger, S. (2007). An approach to classify thermally modified hardwoods by color. Forest Products Journal, 57, 105–110.

Shi, J.L., Kocaefe, D. & Zhang, J. (2007). Mechanical behaviour of Québec wood species heat-treated using ThermoWood process. *Holz als Roh- und Werkstoff*, 65, 255–259.

Sivonen, H., Maunu, S.L., Sundholm, F., Jämsä, S. & Viitaniemi, P. (2002). Magnetic Resonance Studies of Thermally Modified Wood, Holzforschung, 56, 648–654.

Sivonen, H., Nuopponen, M., Maunu, S.L., Sundholm, F. & Vuorinen, T. (2003). Carbon-thirteen cross-polarization magic angle spinning nuclear magnetic resonance and fourier transform infrared studies of thermally modified wood exposed to brown and soft rot fungi, Applied Spectroscopy, 57, 266–273.

Sjöström, E. (1993). Wood chemistry, fundamentals and applications. San Diego, CA: Academic press.

Sundqvist, B., Karlsson, O. & Westermark, U. (2006). Determination of formic-acid and acetic acid concentrations formed during hydrothermal treatment of birch wood and its relation to colour, strength and hardness. Wood Science and Technology, 40, 549–561.

Temiz, A. & Yildiz, U. (2006). The use of modulus of elasticity and modulus of rupture to assess wood decay in laboratory soil-bed test (Doc. No. IRG/WP 06-20338). International Research Group on Wood Preservation.

Tjeerdsma, B.F., Boonstra, M., Pizzi, A., Tekely, P. & Militz, H. (1998). Characterisation of thermally modified wood: molecular reasons for wood performance improvement. Holz als Roh- und Werkstoff, 56, 149–153.

Tjeerdsma, B.F., Stevens, M. & Militz, H. (2000). *Durability aspects of (hydro)thermal treated wood* (Doc. No. IRG/WP 00-40160). International Research Group on Wood Preservation.

Tjeerdsma, B.F. & Militz, H. (2005). Chemical changes in hydrothermal treated wood: FTIR analysis of combined hydrothermal and dry heat-treated wood. Holz als Roh- und Werkstoff, 63, 102–111.

Tuong, V.M. & Li, J. (2011). Changes caused by heat treatment in chemical composition and some physical properties of acacia hybrid sapwood. Holzforschung, 65, 67–72.

Unsal, O. & Ayrilmis, N. (2005). Variations in compression strength and surface roughness of heat-treated Turkish river red gum (*Eucalyptus Camaldulensis*) wood. Journal of Wood Science, 51, 405–409.

Venäläinen, M. Harju, A.M., Kainulainen, P., Viitanen, H. & Nikulainen, H. (2003). Variation in the decay resistance and its relationship with other wood characteristics in old Scots pines. Annals of Forest Science, 60, 409–417.

Vernois, M. (2001). Heat treatment of wood in France – State of the art. In: Review on heat treatments of wood, Proceedings of the special seminar of COST Action E22, Antibes, France.

Viitanen, H., Jämsä, S., Paajanen, L., Nurmi, A. & Viitaniemi, P. (1994). The Effect of Heat Treatment on the Properties of Spruce (Doc. No. IRG/WP 94-40032). International Research Group on Wood Preservation.

Viitanen, H., Metsä-Kortelainen, S., Laakso, T., Iitti, H., Löija, M., Paajanen, L., Harju, A. & Venäläinen, M. (2006). VTT Symposium 244. Applied Material Research at VTT. Symposium on Applied Materials. Espoo, Finland, 8 June.

Viitaniemi P. & Jämsä S. (1996). Puun modifiointi lämpökäsittelyllä [Modification of wood with heat treatment]. Espoo: VTT Publications 814. (In Finnish with English abstract.)

Viitaniemi, P. (1997a). Decay-resistant wood created in a heating process: A heat-treatment process of wood developed by VTT Building Technology yields timber products with enhanced properties. Industrial Horizons, December: 22–23.

Viitaniemi P. (1997b). Thermowood: Modified wood for improved performance. In: Proceedings of the 4th Eurowood Symposium, Wood – The Ecological Material, Stockholm, Sweden (Trätek Rapport No. P9709084, pp. 67–69).

Viitaniemi P., Jämsä S., Vuorinen T., Sundholm F., Paakkari T. & Paajanen L. (2002). Reaction mechanisms of modified wood. In: Paavilainen, L. (editor) & Marttila, S. (assistant editor), Final report, Finnish Forest Cluster Research Programme WOOD WISDOM (1998–2001), Report 3. Helsinki 2002, pp. 185–192.

Virta, J. (2005). Cupping of wooden cladding boards in cyclic conditions: A study of heat-treated and non-heat-treated boards. Building and Environment, 40, 1395–1399.

Wang, J.Y. & Cooper, P.A. (2005). Effect of oil type, temperature and time on moisture properties of hot oil-treated wood. Holz als Roh- und Werkstoff, 63, 417–422.

Weiland, J.J. & Guyonnet, R. (2003). Study of chemical modifications and fungi degradation of thermally modified wood using DRIFT spectroscopy. Holz als Roh- und Werkstoff, 61, 216–220.

Welzbacher, C.R. & Rapp, A.O. (2005). Durability of different heat treated materials from industrial processes in ground contact (Doc. No. IRG/WP 05-40312). International Research Group on Wood Preservation.

Welzbacher, C.R. & Rapp, A.O. (2007). Durability of thermally modified timber from industrial-scale processes in different use classes: Results from laboratory and field tests. *Wood Material Science and Engineering*, 2, 4–14.

Welzbacher, C.R., Brischke, C. & Rapp, A.O. (2007). Influence of treatment temperature and duration on selected biological, mechanical, physical and optical properties of thermally modified timber. *Wood Material Science and Engineering*, 2, 66–76.

Wikberg, H. & Maunu, S.L. (2004). Characterisation of thermally modified hard- and softwoods by ^{13}C CPMAS NMR. Carbohydrate Polymers, 58, 461–466.

Wise, L.E. & Jahn, E.C. (1952). Wood Chemistry. Reinhold publishing company, United States of America.

WTT. (2011). WTT Wood treatment technology. http://www.wtt.dk/Hear4t_Treatment.asp. Accessed Apr 2011.

Yildiz, S., Gezer, D. & Yildiz, U.C. (2006). Mechanical and chemical behavior of spruce wood modified by heat. Building and Environment, 41, 1762–1766.

Zaman, A., Alén, R. & Kotilainen, R. (2000). Thermal behaviour of Scots pine (Pinus sylvestris) and Silver birch (Betula pendula) at 200–230ºC. Wood and Fiber Science, 32, 138–143.

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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 170ºC, 190ºC, 210ºC and 230ºC. 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 (230ºC) 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 (190–210ºC), 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)
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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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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.

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