CHARACTERISTICS OF WOOD PLASTIC COMPOSITES BASED ON MODIFIED WOOD

- MOISTURE PROPERTIES, BIOLOGICAL RESISTANCE AND MICROMORPHOLOGY
CHARACTERISTICS OF WOOD PLASTIC COMPOSITES BASED ON MODIFIED WOOD - MOISTURE PROPERTIES, BIOLOGICAL RESISTANCE AND MICROMORPHOLOGY

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Doctoral Thesis
All aboard!

Ozzy
ABSTRACT

Biobased materials made from renewable resources, such as wood, play an important role in the sustainable development of society. One main challenge of biobased building materials is their inherent moisture sensitivity, a major cause for fungal decay, mold growth and dimensional instability, resulting in decreased service life as well as costly maintenance. A new building material known as wood-plastic composites (WPCs) has emerged. WPCs are a combination of a thermoplastic matrix and a wood component, the former is usually recycled polyethylene or polypropylene, and the latter a wood processing residual, e.g. sawdust and wood shavings.

The objective of this thesis was to gain more insight about characteristics of WPCs containing a modified wood component. The hypothesis was that a modified wood component in WPCs would increase the moisture resistance and durability in outdoor applications. The study comprises both injection molded and extruded WPC samples made with an unmodified, acetylated, thermally modified or furfurylated wood component in a polypropylene (PP), high density polyethylene (HDPE), cellulose ester (CAP, a cellulose ester containing both acetate and propionate substituents) or polylactate (PLA) matrix. The WPCs were prepared with 50-70 weight-% wood. The emphasis was on studying the moisture sorption, fungal resistance and micromorphological features of these new types of composites. Water sorption in both liquid and vapor phases was studied, and the biological performance was studied both in laboratory and in long term outdoor field tests. Micromorphological features were assessed by analyzing of the wood component prior to and after processing, and by studying the composite microstructure by means of a new sample preparation technique based on UV excimer laser ablation combined with scanning electron microscopy (SEM).

Results showed that the WPCs with a modified wood component had a distinctly lower hygroscopicity than the WPCs with unmodified wood, which resulted in less wood-plastic interfacial cracks when subjected to a moisture soaking-drying cycle. Durability assessments in field and marine tests showed that WPCs with PP or CAP as a matrix and 70 weight-%
unmodified wood degraded severely within a few years, whereas the corresponding WPCs with a modified wood component were sound after 7 years in field tests and 6 years in marine tests. Accelerated durability tests of WPCs with PLA as a matrix showed only low mass losses due to decay. However, strength losses due to moisture sorption suggest that the compatibility between the PLA and the different wood components must be improved. The micromorphological studies showed that WPC processing distinctly reduces the size and changes the shape of the wood component. The change was most pronounced in the thermally modified wood component which became significantly reduced in size. The disintegration of the modified wood components during processing also creates a more homogeneous micromorphology of the WPCs, which may be beneficial from a mechanical performance perspective. Future studies are suggested to include analyses of the surface composition, the surface energy and the surface energy heterogeneity of both wood and polymer components in order to tailor new compatible wood-polymer combinations in WPCs and biocomposites.

**Keywords:** Wood plastic composites, WPC, acetylation, thermal modification, furfurylation, moisture sorption, biological durability, UV excimer laser, micromorphology.
SAMMANFATTNING (IN SWEDISH)

Biobaserade material tillverkade av förnybara råvaror spelar en betydande roll för en hållbar samhällsutveckling. En utmaning för biobaserade byggnadsmaterial är deras inneboende fuktkänslighet, som ökar risken för biologiskt angrepp, mögelpåväxt och dimensionsförändringar, vilket i sin tur kan leda till en nedsatt livslängd och kostsamt underhåll. En ny typ av byggnadsmaterial, trä-plastkompositer (eng. wood plastic composites, WPCs), finns nu på byggnadsmaterialmarknaden i Sverige. WPCs är en kombination av en träkomponent, till exempel i form av träbearbetningsrester såsom sågspån eller hyvelspån, och en termoplastisk matris ofta i form av återvunnen polyeten eller polypropen.

Syftet med denna avhandling har varit att få en djupare insikt om några egenskaper hos WPCs med en modifierad träkomponent. Hypotesen var att en modifierad träkomponent i WPCs skulle minska fuktkänsligheten och samtidigt öka den biologiska beständigheten för tillämpningar utomhus. Studien innefattar både formsprutade och extruderade WPC-material med en träandel mellan 50 och 70 vikts-%. Träkomponenterna bestod av omodifierade, acetylerade, termiskt modifierade eller furfuryleferade träspån eller fibrer. Matrismaterialen bestod av polypropen (PP), högdensitetspolyeten (HDPE), cellulosaester (CAP, en cellulosaester innehållande både acetat och propionat substituent) eller polylaktat (PLA). Tonvikten har lagts på att studera fuktegenskaper, resistens mot rötangrepp och mikromorfologi hos dessa nya typer av kompositer. Mikromorfologin studerades genom analys av träkomponenten före och efter bearbetning samt genom studier i svepelektromikroskop (SEM) på ytor beredda med en ny provberedningsmetod baserad på ablatering med UV-excimerlaser.

Resultaten visade att WPCs med en modifierad träkomponent hade en betydligt lägre hygroskopicitet än WPCs med en omodifierad träkomponent, vilket minimerade gränssiktsprickor mellan trä och plast då kompositerna utsattes för en uppfuktning-uttorkningscykel. Beständighetsutvärdering i fältförsök ovan och i mark, samt i marin miljö, visade att WPCs med 70 vikts-% modifierad träandel har klarat sig utan biologiska
angrepp efter 7 år i fältförsöken och efter 6 år i de marina försöken, medan motsvarande WPCs med en omodifierad träkomponent uppvisade kraftig nedbrytning redan efter några år. Accelererad beständighetsutvärdering av WPCs med PLA som matris påvisade inga rötagrepp, emellertid så sänktes hållfastheten på grund av fuktsorption vilket tyder på att kompatibiliteten mellan PLA och de olika träkomponenterna bör förbättras. De mikromorfologiska studierna visade att tillverkningsprocessen för WPC-materialen drastiskt minskar storleken och ändrar formen hos träkomponenten. Förändringen var störst för den termiskt modifierade träkomponenten som kraftigt reducerades i storlek under processen. Förändringen av de modifierade träkomponenterna under processen resulterade i en mer homogen mikromorfologi, vilket kan ge förbättrade mekaniska egenskaper hos WPC-materialen. Framtida studier föreslås omfatta analyser av ytkemisk sammansättning, ytenergi och ytons heterogenitet hos både trä- och polymerkomponenten för att kunna skräddarsy nya kompatibla trä-polymerkombinationer i WPCs och biokompositer.
PREFACE

This work has been carried out at Kungliga Tekniska Högskolan, avdelningen för Byggnadsmaterial (KTH Royal Institute of Technology, Division of Building Materials), Stockholm, Sweden. The work has been a part of the Institute Excellence Centre EcoBuild, hosted by SP Wood Technology in Stockholm. The over-all objective of this centre is to engineer and develop innovative, eco-efficient and durable wood based materials and products for building, furniture and textile applications, see also www.ecobuild.se for further information. Types of materials of primary interest are: modified solid wood such as thermally modified, furfurilated and acetylated wood, biobased textile fibres, biobased binders and coatings, solvent-spun cellulose fibres and biocomposites. A top priority effort is to engineer fully biobased alternative products entirely based on renewable resources. EcoBuild is mainly financed by VINNOVA (Swedish Governmental Agency for Innovation Systems), SSF (Swedish Foundation for Strategic Research) and the Knowledge Foundation and together with contributions from the participating companies.

An acknowledgement is addressed to FORMAS for their financial support (Dnr 24.3/2003-0690) regarding the development of the UV laser technique for sample preparation used in this work and their financial support (Dnr 243-2007-1166) regarding acquisition of the scanning electron microscope used in this work, as well as the Knut and Alice Wallenberg foundation (Dnr KAW 1998.0130) for funding the UV laser laboratory.

I wish to express my sincere gratitude to my supervising group at KTH and SP Wood Technology consisting of main supervisor Prof. Magnus Wålinder, Dr. Pia Larsson Brelid, Dr. Mats Westin and Prof. em. Ove Söderström for their enthusiasm and support.

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Stockholm, November 2012

Kristoffer Segerholm
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This doctoral thesis is based on the following papers, referred to in the text by their roman numerals:


Other relevant publications not included in this thesis:


OUTLINE OF THE THESIS

Chapter 1 presents the general context of the thesis, a short background of wood plastic composites and the chemical modification of wood using acetylation, thermal modification and furfurylation, and finally the statement of objectives.

Chapter 2 describes the materials manufactured and evaluated in this thesis.

Chapters 3, 4 and 5 summarize and discuss the experiments and results of the moisture sorption, the biological durability and the micromorphology studies.

Finally, Chapter 6 presents conclusions that can be drawn from the work in this thesis as well as some suggested future activities related to this work.
1. INTRODUCTION

1.1 WOOD PLASTIC COMPOSITES

New types of biobased building materials made from renewable resources, such as wood, are becoming more interesting. The main reason for this can be related to global environmental challenges and the need for a sustainable development in society. There is a long tradition of using thermoplastic materials, such as polyethylene and polypropylene, and an even a longer tradition of using wood-based materials, such as particleboards and fiberboards. A new material has emerged, which is a combination of a thermoplastic component and a wood based component, known as wood-plastic composites (WPCs). The term wood-plastic composites is historically a broad term that includes all wood containing composites in both a thermoset or a thermoplastic matrix. In this thesis, only wood-thermoplastic composites are considered, and the term WPCs will be used throughout this thesis for this type of material.

To produce WPCs, wood residuals, e.g. sawdust, wood shavings or wood flour, are mixed with a thermoplastic polymer and melt processed (or thermoformed) into its final shape, either as a continuous profile forming through extrusion or as a three dimensional form through injection molding. During recent decades, WPCs have rapidly increased their market share as a building material (Carus and Gahe 2008). Despite the recent economic downturn, which has leveled off this growth, a continued global market advance is predicted (Anonymous 2011). This success may partly be attributed to the fact that WPCs are a competitive alternative to tropical hardwoods and that they are considered to require less maintenance than conventional wood products. Using WPCs it is also possible to manufacture more complex shapes than with solid wood products, with a raw material yield close to 100%.
Figure 1. WPC cable channel lid manufactured by OFK Plast AB, Karlskoga, Sweden.

The initial growth of WPCs as a building material was in the decking market (Clemons 2002), other profile products, for example sidings, fencing, and piling also exists to a lesser extent. Three dimensional forming of WPCs by injection molding allows for more freedom when designing the product, and the product can be finalized in one processing step. Figures 1 and 2 demonstrate two innovative products made out of WPC material. Figure 1 shows a cable channel lid made of WPC used to cover cable channels along the railroads, and Figure 2 demonstrates a new type of injection molded pallet that meets the mechanical requirements of EU pallets. The pallet is designed with a fit function that enables stacking at a height that is less than the sum of the height of the individual pallets, and consequently, lower than the stacking height of conventional pallets.

Some advantages of WPCs compared with glass fiber reinforced or mineral filled thermoplastics are less environmental impact (e.g. lower embedded energy, smaller carbon footprint, and better recyclability), a less abrasive processing, lower price, increased cooling rate (leading to a decrease in product cycle time in injection molding) (Youngquist 1995),
and in addition, lower thermally induced movements than unfilled plastics. From the same perspective, some disadvantages of WPCs are that the wood component adds the risk of moisture sorption and biological degradation, and the necessity of maintaining a lower temperature during processing due to the risk for severe thermal degradation of the wood polymers. Another main challenge of WPCs is their more brittle behavior, both in comparison with solid wood and glass fiber reinforced plastics. Compared with conventional unmodified and preservative treated solid wood, WPCs are more expensive to produce and have a lower strength to weight ratio.

![Figure 2](image_url)

**Figure 2.** Injection molded WPC pallet, design IKEA of Sweden. Construction currently not in use.

The wood component used in conventional WPCs often originates from planer shavings or sawdust. The producers of WPCs normally use commercial wood flour, which has a broad size distribution, and consequently makes it more difficult to predict the properties of the WPC products. Typical particle sizes used in WPCs are 10-80 mesh (Clemons 2002). In a comparative study on the effects of particle shape, Stark and Rowlands (2003) have concluded that it is the particle shape, not the size that has the greatest influence on strength and stiffness. A more slender particle will redistribute and transfer stresses better between the particles.
and the matrix. Therefore, the process for wood particle preparation should be designed to give particles with high length to width ratio. However, the size distribution may also be of great importance. For indoor products, the challenge is mainly to achieve sufficient mechanical properties of the material for the intended product, since better mechanical properties give more freedom for the design of a product.

Outdoor durability of WPCs

In the case of outdoor use of WPCs, the challenges are related to the requirements of being a long lasting and minimum maintenance material. Exposure to the outdoor environment implies moisture and temperature fluctuations, the risk of attack by micro-organisms and degradation by UV-radiation. The moisture properties, dimensional stability and biological durability of WPCs are highly dependent on the constituents and the processing of the material. If the wood component is allowed to sorb moisture, the risk of irreversible dimensional changes arises, i.e. wood-polymer delamination, and the risk of degradation by fungi and other micro-organisms also arises.

WPC manufacturers often promote their products as maintenance-free and highly durable with a lack of cracking and splintering, often offering 10-year warranties (Clemons 2002). However, the first generation of WPCs has shown to lack in long time durability and failures have led to class action law suits (Morris and Cooper 1998). Studies on WPCs and moisture transport have shown that the initial moisture content (MC) is very low and the rate of sorption is very slow. However, the moisture in the composites is not uniformly distributed and the outermost part can reach very high moisture levels, high enough to support fungal degradation (Gnatowski 2009, Wang and Morrell 2004). It is also important to stress that even if moisture uptake is slow in WPCs, even when immersed in water, the uptake may continue over a long period of time. The rate and extent of moisture sorption increase when the wood content exceeds approximately 50% of the total weight of the composite (Clemons et al. 2012). In addition, a moist environment will swell the wood particles close to the surface, and the particles will shrink upon drying. This will cause stresses within the material and create microcracks, which will expose
more particles deeper in the material. This swelling and shrinkage will also cause cracks at the interfaces between the wood particles and the matrix.

Laboratory soil block tests have shown mass losses of 3-8% due to the fungal degradation of WPCs after 12 weeks of exposure (Ibach et al. 2003, Clemons and Ibach 2002). The mechanical durability of WPC products has been evaluated for WPC products for use in marine waterfront applications, and it was concluded that moisture ingress into the composite can reduce strength up to 50% (Smith and Pooler 2000). Improvements are needed to minimize these problems, and moisture in WPCs with a high loading of the wood component is definitely one of the key reasons for their shortcoming in both mechanical durability and resistance to fungal decay.

**WPCs with modified wood**

A chemically modified wood component can be introduced into the composite which may increase both dimensional stability and resistance to biological attack (see e.g. Segerholm 2007, Ibach and Clemons 2006). This would improve durability and resistance to decay.

Several environmentally friendly methods for the chemical modification of wood have been developed, most of them are not new, but have previously failed to gain commercial interest. Three methods that have recently have been commercialized are acetylation, furfurylation and thermal modification (see e.g. Jones et al. 2012, Hill et al. 2010, Englund et al. 2009, Hill et al. 2007). Acetylation of wood was mentioned as early as in 1928, where acetylation was used in a procedure for isolating lignin (Fuchs 1928). Early work on the acetylation of solid wood in order to improve its dimensional stability was performed by Stamm and Tarkow in 1947 and in 1950 (Stamm and Tarkow 1947, Tarkow et al. 1946). Research pertaining to furfurylation was initiated by Stamm in the early 1950s. Most of the early work was performed by his student Goldstein (cf. e.g. Goldstein and Dreher 1960, Goldstein 1955). Some of the scientific reports on thermal modification of wood goes as far back as to Tiemann in 1915 (Hill 2006), who discovered that thermally modified wood showed reduced moisture sorption with only small reductions in strength. The reduced moisture
sorption of thermally modified wood depends on the initial thermal degradation of the hemicelluloses (Stamm 1964), which are the most hygroscopic constituents in the wood. These modification methods have been extensively studied in the literature, and several improvements have been made since the work was initiated. These and other modification methods are thoroughly described in the recent book by Hill (2006). Figure 3 illustrates two demonstrator products made from modified wood.

Abdul Khalil et al. (2002) have shown that thickness swelling and moisture levels of WPCs can be reduced by using acetylated *Acacia magnum* as a wood component. The amount of wood in that study was between 20 and 50% by weight. Resistance to biological decay has been studied in laboratory tests, both in single fungi test jars and in soil boxes with a variety of different fungi and micro-organisms (Westin et al. 2006, Ibach et al. 2003, Clemons and Ibach 2002), as well as in field and marine tests. Current standards for fungal resistance have been developed for testing solid wood (see e.g. ENV 807, EN 252, EN 113), however, the slow moisture uptake of WPCs makes these standard test methods inappropriate for use directly, and modifications of such test procedures are needed. Pre-treatment to increase the initial moisture content has been used, but remains to be further refined (Defoirdt et al. 2010, Van Acker 2006, Ibach et al. 2003, Clemons and Ibach 2002).

In the present investigation, the conceptual idea was to use residuals from the production of modified wood or fibers, such as sawdust, shavings or boards rejected because of cracks or discoloration. This will mean that no additional wood resources were used and the waste products were turned into value added products. An increase in the resistance of the wood component to moisture and fungal decay could enable a significantly higher weight-% wood in WPCs for outdoor use, which could result in a lower overall cost of the composite because of less use of the generally more expensive thermoplastic matrix.
1.2 STATEMENT OF OBJECTIVES

The objective of this thesis was to study some characteristics of wood plastic composites that incorporate an acetylated, a thermally modified or a furfurylated wood component in a polypropylene, a polyethylene, a cellulose ester or a polylactate matrix to gain knowledge about the behavior of such materials in a long term perspective. The emphasis has been on studying:

- Moisture properties of these composites both in liquid and vapor phases.
- Biological performance both in laboratory tests and long term outdoor field tests.
- Micromorphological features of the WPCs and mechanical damage done to the wood component during processing.
2. MATERIALS PREPARATION

All the WPCs prepared in this work contained at least 50 weight-% (wt.-%) wood, and the extruded WPC materials contained 60-70 wt.-% wood. Such a high wood content entails challenges both to dispersing the wood components during processing and also to protecting the WPCs against moisture sorption, moisture induced movements and biological degradation. The primary idea in this thesis was to develop a new type of durable high-wood-content WPCs containing a chemically modified wood component, that exhibit superior moisture resistance, dimensional stability and resistance to biological degradation.

2.1 MODIFIED WOOD

A brief description of the modified wood used in this thesis is given in this section. The unmodified wood used in the thesis was prepared from Scots pine (*Pinus silvestris*) sapwood boards.

*Acetylation with acetic anhydride*

Modification of wood by acetylation is a single-site reaction where one acetyl group replaces one hydroxyl group in the cell wall polymers. In the modification procedure, the wood material is impregnated with acetic anhydride and then reacted at an elevated temperature (Rowell 2006a). The only by-product produced is acetic acid. The resulting modified wood material exhibits increased dimensional stability, maintained strength, decreased equilibrium moisture content (EMC), and superior resistance to biological degradation (Larsson Brelid 1998).

In this thesis, acetylated Scots pine (*Pinus silvestris*) sapwood boards were prepared by A-Cell Acetyl Cellulosics AB in their pilot plant (now located at SP, Borås, Sweden) with a 0.66 m³ reactor (Figure 4), according to a simplified procedure without the use of any catalyst or co-solvent in the reaction (Larsson Brelid 1998, Rowell *et al.* 1986). The acetylation level of the wood material used in this study was approximately 18-23% expressed as wood acetyl content. The acetylation of fibers was conducted in the
liquid phase by BP Chemicals in 100 kg batches, and the degree of acetylation was about 20% expressed as wood acetyl content.

Figure 4. Microwave heated wood acetylation pilot plant reactor situated in the EcoBuild laboratory at SP, Borås, Sweden. Left: overview, Right: inside the cylinder.

Thermal modification

Thermal modification of wood results in a change in color and a partial degradation and rearrangement of the cell wall polymers. The hemicelluloses are the most affected constituents (Rowell et al. 2009). It is important to note that the thermal treatment of resinous wood species also results in modification and a spatial redistribution of the wood extractives (Nuopponen et al. 2003). The resulting material exhibits a higher dimensional stability, a reduced hygroscopicity and improved resistance to microbial decay, but has reduced mechanical properties. There are four major heat treatment methods used in Europe today; ThermoWood, Oil Heat Treatment, Plato Wood and Retification. These four are similar in that solid wood is subjected to a temperature of around 200 °C for several hours in a low oxygen atmosphere (Rapp 2001). In this thesis, thermally modified Norway spruce (Picea abies) boards were prepared according to
the ThermoWood D (Anonymous 2003) procedure. The process has a peak temperature of 212 °C.

Furfurylation

So-called furfurylation of wood involves treatment with furfuryl alcohol, which is pressure impregnated into the wood and then *in-situ* polymerized within the cell wall using elevated temperature and catalysts (Lande 2008). There are also strong indications that the furfuryl alcohol initially reacts with the cell wall lignin and that the formed furan polymer is thereby chemically bonded to the cell wall (Nordstierna *et al.* 2008). The resulting material exhibits high dimensional stability, improved mechanical behavior, except for impact resistance, and improved resistance to fungal decay (Lande *et al.* 2004). In this thesis, furfurylated radiata pine (*Pinus radiata*) boards were prepared according to Lande *et al.* (2004) in an industrial pilot plant of former Wood Polymer Technologies ASA (WPT), now Kebony ASA, Skien, Norway. Results related to WPCs with a furfurylated wood component are included in Paper VII.

2.2 PREPARATION OF WOOD COMPONENTS

In Papers II-VII ground wood was prepared from the modified and unmodified solid wood samples using a two-step grinding process involving first a disk flaker to produce thin veneers, followed by a knife ring mill to produce the finer wood components (Figure 5). The size and shape of the wood component vary depending on the modification of the solid wood prior to grinding; thermally modified wood disintegrates into very fine particles with minor variation in size, whereas the acetylated and unmodified wood disintegrates into larger particles with a greater variation in size. Paper VI also involved liberated softwood fibers, medium density fiberboard (MDF) type fibers, which were used as they were or modified through acetylation.
Figure 5. Left: disk flaker for preparation of thin flakes below. Right: knife ring mill for preparation of the wood component for WPCs.

2.3 POLYMER MATRICES

Thermoplastic matrices for the use in WPCs need to be processable at temperatures below 200 °C, since wood constituents start to thermally degrade at approximately 150 °C, and around 200 °C the degradation may be substantial for many wood species (Fengel and Wegener 1983). Thermal degradation is also dependent on the wood-plastic residence time at higher temperatures, i.e. if the processing cycle is kept short it is possible to use a higher peak temperature. Thermoplastic matrices currently used in commercial WPCs are normally high density polyethylene (HDPE), polypropylene (PP) or polyvinyl chloride (PVC). Other matrices that have gained increased interest in recent years are so-called bioderived plastics, e.g. cellulose esters and polylactates, which are made partly or fully from renewable resources.

This thesis comprises work with both traditional olefin thermoplastics such as polypropylene (PP) and high density polyethylene (HDPE), and
bioderived matrices, such as cellulose acetate propionate (CAP) and polylactate (PLA). Both CAP and PLA are able to sorb moisture, and therefore, need to be pre-dried before processing.

2.4 MANUFACTURING OF WPC SAMPLES
The two main techniques for manufacturing WPCs are injection molding and extrusion. Normally, a physical mixture of wood components, plastic and additives is dry blended and compounded on an extruder to achieve WPC granules prior to injection molding or extrusion. In injection molding, the granules are melted and injected into a mold to give the detail its final shape. In extrusion the granules are melted and pushed through a die, which will gives the profile its shape.

In this thesis, WPC materials with wood and matrix components as described in Sections 2.2 and 2.3 were prepared by either injection molding or extrusion. In some cases processing additives were added to the formulation. Figure 6 illustrates the different WPC materials used in Papers II–VII. Papers III and VI involve injection molded WPC samples with a cross section of 4 x 10 mm² (Figures 6d and e), Paper IV involves extruded WPC profiles with a cross section of 3 x 13 mm² (Figure 6c), Papers II, V and VII involve extruded hollow WPC profiles with a cross section measuring 60 x 40 mm² with a wall thickness of 8 mm prepared using a conical extruder (Conex®) (Figure 6a), and Paper VII also involves extruded decking profiles with a cross section measuring 140 x 25 mm² (Figure 6b). As mentioned in Section 2.1, WPCs with a furfurylated wood component were only included in Paper VII. This is due to extrusion processing difficulties, which meant that only a very limited amount of the composites were considered acceptable for inclusion in the further research studies.
Figure 6. WPC materials used in the thesis: a) Extruded hollow profile, b) Extruded solid decking profile, c) Extruded 3 × 13 mm² profile, d) Injection molded tensile test bar, e) Injection molded bend test bar (dark color due to thermally modified wood component).
3. MOISTURE CHARACTERISTICS

Moisture intrusion in WPCs directly affects the dimensions of the material due to the swelling forces of the wood component. Indirectly, if the material is able to sorb moisture, it will be more susceptible to decay by fungi and other micro-organisms. In WPCs with PP and HDPE, the polymer matrix constitutes a hydrophobic phase and the wood component a hydrophilic phase. Because of this, the moisture transport into the material mainly takes place through the wood component and internal void spaces in the material. When CAP and PLA are used, the matrices are able to sorb moisture and, thus, also contribute to the moisture transport into the material. The surfaces of WPCs are usually smooth, and a thin surface layer generally consists of a high proportion of the polymer matrix. In the initial stage of moisture exposure, this polymer-rich surface phase retards the rate of moisture sorption. Therefore, the WPCs will appear to be very moisture resistant. However, when the composite is subjected to UV radiation and/or water, this surface layer may be degraded, resulting in a decreased moisture resistance, and a critical intrusion of water into the bulk of the material may occur more easily.

3.1 WATER VAPOR SORPTION

In Papers III-VI the water vapor sorption properties of the WPC materials have been evaluated. Thin specimens were used in these studies; a thickness of 2 mm in Papers III and V and 1 mm in Paper IV. All surfaces were sealed except for the original outer surface of the WPCs. In Paper VI, the inlet piece for the injection molded bending test bars was used for equilibrium moisture content measurements. The moisture sorption rate was very slow for all the WPC materials, especially slow for the injection molded WPCs due to the lower amount of wood component in those composites compared to the extruded WPCs. In Paper III for WPCs with PP the test was ended after 18 months, and in Paper VI for WPCs with PLA as matrices the test was ended after 21 months. In both tests the specimens had still not reached their equilibrium moisture content (EMC) at the end of the tests. However, in Paper III, another set of the WPCs with PP as the matrix was subjected to artificial weathering prior to the
water vapor sorption test, and in that case the specimens reached EMC in much shorter times (approximately 120 days). In addition, composites with CAP as the matrix were evaluated in Paper III, and due to the ability of CAP to sorb and transport moisture, the times to reach EMC were much shorter and no difference was observed between non-aged and pre-aged specimens.

In Paper IV, WPC samples subjected to artificial ageing procedures involving weathering, and white- and brown-rot decay (Table 1) were evaluated in dynamic vapor sorption experiments. Figure 7 shows the moisture uptake of WPC samples with an unmodified wood component when the climate was changed from a conditioned state at 65% relative humidity (RH) to a climate with 90% RH at a constant temperature of 27 °C. As can be seen, the artificial weathering of the specimens changed the rate of moisture sorption substantially compared to the non-weathered samples. The WPCs with an acetylated wood component were also affected by the artificial weathering giving higher sorption rates than the non-weathered samples. However, the rate and level of moisture gain was much lower for the WPCs with an acetylated wood component than it was for the WPCs with an unmodified wood component (Figures 7 and 8).

**Table 1. Artificial ageing procedures used for the WPC samples with 50 wt.-% wood component in Paper IV**

<table>
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<th>Artificial ageing procedure</th>
<th>In Figure</th>
</tr>
</thead>
<tbody>
<tr>
<td>U1, A1</td>
<td>Room temperature (22°C) water soaking for 2 weeks</td>
<td>7</td>
</tr>
<tr>
<td>U2, A2</td>
<td>Artificial weathering for 1000 hours, followed by room temperature (22°C) water soaking for 2 weeks</td>
<td>7</td>
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<tr>
<td>U3, A3</td>
<td>Artificial weathering for 1000 hours and room temperature (22°C) water soaking for 2 weeks, followed by a modified soil block test procedure based on ASTM D1413 with <em>Trametes versicolor</em>, a white-rot fungus</td>
<td>7</td>
</tr>
<tr>
<td>U4, A4</td>
<td>Artificial weathering for 1000 hours and room temperature (22°C) water soaking for 2 weeks, followed by a modified soil block test procedure based on ASTM D1413 with <em>Gloeophyllum trabeum</em>, a brown-rot fungus</td>
<td>7</td>
</tr>
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Figure 7. Moisture uptake versus square root of time when relative humidity is raised from 65% to 90% for extruded WPC specimens with 50 wt.-% unmodified wood component.

Figure 8. Moisture uptake versus square root of time when relative humidity is raised from 65% to 90% for extruded WPC specimens with 50 wt.-% acetylated wood component.
The rate of sorption is, to a large extent, governed by the polymer matrix and the wood content in the WPCs. However, the modifications by acetylation, furfurylation or thermal treatment of the wood component have a substantial effect on the amount of moisture that can be sorbed by the material. By using an acetylated wood component in the WPCs, the levels of moisture sorbed can be reduced to at least 50% of that for the WPCs with unmodified wood. Such a reduction in moisture sorption in the material also has an effect on the dimensional stability of the material.

3.2 WATER SOAKING

Water soaking experiments were performed, both to precondition the specimens to be evaluated regarding biological durability and to study micromorphological changes due to moisture sorption.

In Paper V, specimens for biological testing were preconditioned by water soaking. Specimens measuring 5 x 10 x 100 mm\(^3\) were cut from the extruded hollow profiles with approximately 70 wt.-% wood. The water soaking resulted in very high moisture contents (MC), for the extruded WPC samples with unmodified, acetylated and thermally modified wood components after a total soaking time of 4 weeks (Table 2). On the other hand, in Paper VI the injection molded WPC samples with 50 wt.-% wood and PLA matrix subjected to 2 weeks of soaking only reached 3.7% MC for the sample with unmodified wood. The MC for the WPC samples with a modified wood component was even lower.

<table>
<thead>
<tr>
<th>Table 2. Moisture content (MC) before and after soaking in water for extruded WPC specimens with 70 wt.-% wood component.</th>
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<tbody>
<tr>
<td><strong>WPC material</strong></td>
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<tr>
<td>PP/unmodified pine</td>
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<tr>
<td>PP/acetylated pine</td>
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<tr>
<td>PP/thermally modified spruce</td>
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</table>
4. BIOLOGICAL DURABILITY

There are three main types of wood degrading fungi, white-, brown-, and soft-rot. White rot preferentially degrades lignin, brown rot mainly attacks hemicelluloses and cellulose, and soft rot degrades all cell wall polymers (Eaton and Hale 1993). A criterion for these fungi to attack wood is that moisture within the material reaches a certain level. As described in Chapter 3, the moisture transport in WPCs is very slow. Consequently, the time for gaining sufficient moisture suitable for attack by decaying fungi is much longer for WPCs than for solid wood. Another factor influencing the biological durability of WPCs is the amount of wood in the composites. At higher levels (above about 50%), the wood component may form an interconnecting network in the matrix which accelerates the moisture transport into the material. This both affects the biological durability and the dimensional stability of the composite.

4.1 LABORATORY TESTING

The laboratory testing included in this thesis was performed using a terrestrial microcosm (TMC) test according to an extended version of ENV 807. Prior to the TMC test the specimens were subjected to preconditioning involving a 2 week leaching procedure in de-ionized water, according to EN 84, followed by a 14-day water soaking in de-ionized water and then direct insertion into the test soils. Scots pine (Pinus silvestris) control specimens were used to follow the fungal activity in the soils. Three different soils were used in the TMC tests, TMC1 was a compost soil with a high activity of soft rot, TMC2 was a soil from the Simlångsdalen test field with predominately brown-rot decay, and TMC3 was a forest soil with high activity of white-rot decay. The specimens were buried three quarters of their lengths in the soil, Figure 9 shows the experimental setup and a schematic drawing of the specimens inserted in the soil. After the test, the specimens were cleaned and dried, and after weighing the mass losses were calculated. The corrected mass loss, solely caused by decay was calculated by subtracting mass loss due to leaching from specimens from sterile soils.
Extruded profiles with 70 wt.-% modified wood are evaluated in the TMC test in Paper V. Specimens 5 x 10 x 100 mm$^3$ were cut out from the extruded profiles in such a way that the outer polypropylene (PP)-rich surface was removed. The findings showed that the WPC control sample with unmodified wood had mass losses due to decay. The WPCs with thermally modified wood also had slight mass losses, but decay could not be verified by microscopy. Mass losses for WPCs with acetylated wood were very low and no decay could be detected by microscope. Figure 10 shows the corrected mass losses after 32 weeks of exposure to the test soils.
Figure 10. Corrected mass loss after 32 weeks exposure to three different TMCs for extruded WPCs with 70 wt.% of unmodified, thermally modified or acetylated wood component as well as for pine solid wood and PP control specimens.

Injection molded WPCs are evaluated in Paper VI. The wood components used were both in the shape of fibers and also in the shape of particles. The thermoplastic used in this study was a polylactate (Nature Works R 4042D, NatureWorks LLC, Minnesota, USA) which originates from renewable resources and is possible to compost at the end of life. Tensile test bars with 50 wt.-% were prepared, the specimens measured 4 x 10 x 80 mm³, no further processing of the WPCs before the biological testing were needed. The biological test resulted in very low mass losses due to decay, however, the mechanical evaluation before and after the biological test showed major differences. The mechanical evaluation showed that the PLA unmodified wood fiber composites lost great amounts in strength and stiffness, this was mainly due to moisture-induced movements of the wood component and not due to biological decay.
4.2 FIELD TESTING

In field tests, materials are subjected to an environment which, in comparison to laboratory experiments, represents the real world. However, field tests are very time-consuming and several years may pass before any detectable decay occurs, and an even longer time may pass before there is substantial decay in the tested materials. The correlation between results from laboratory experiments and field tests is often difficult to do, especially for new types of materials. This is true for WPCs for which a limited number of field test studies of biological durability have been conducted.

The field and marine tests are evaluated in Paper VII. Extruded profiles with PP and CAP have been in a horizontal double layer test (Figure 11) and in ground tests (EN 252, Figure 12) for 7 years and in a marine test (EN 275) for 6 years. The samples in each test have been evaluated annually and remain in the field and marine sites.

Figure 11. Double layer test set up.
Figure 12. *In ground test set up.*

**Horizontal double layer**

In the horizontal double layer test specimens have been placed horizontally in two layers on top of each other with a half width shift of the upper layer to the lower layer, the specimen size is 50 x 25 x 500 mm$^3$ except for the extruded hollow profiles which were only cut in length to 500 mm in length. The rig has been built so that the specimens can be placed 200 mm above ground level. The samples with an unmodified wood component showed major dimensional changes due to moisture pick-up at an early stage, and all stakes had a substantial index of decay after 7 years of testing in the horizontal double layer. The samples with a modified wood component still perform very well and are still considered to be sound. Figure 13 shows three extruded WPC stakes from the double layer test after 7 years of exposure. The bottom stake has been made with an unmodified wood component and shows severe growth of fungal mycelium.
Figure 13. Three extruded WPC stakes with PP matrix and 70 wt.-% wood from the double layer test after 7 years of exposure. From top to bottom; thermally modified, acetylated and unmodified wood component.

In ground test

In the in ground test, stakes have been buried to half their length in soil. The specimen size is 50 x 25 x 500 mm$^3$ except for the extruded hollow profiles which were only cut in length to 500 mm in length. The findings from the test sites in Borås and Simlångsdalen are presented in Paper VII. In the in ground test, the degradation of the materials has been at a higher rate than in the horizontal double layer. All the stakes with unmodified wood component show decay. Some of the stakes with modified wood components show signs of decay, but are still considered to be sound. Figure 14 shows two extruded WPC profiles with an unmodified and an acetylated wood component. The right part of the stakes has been above ground and the left part in the ground. The white appearance is due to UV-degradation and the swollen and twisted appearance of the upper stake is due to moisture induced movements of the unmodified wood component.
Figure 14. Two extruded WPC stakes with PP matrix and 70 wt.-% wood from the in ground test after 7 years of exposure, top stake with unmodified wood component and bottom stake with acetylated wood component.

Marine test

Marine testing of WPC specimens is being conducted according to EN 275. The test specimens measuring 50 x 25 x 200 mm³ with a hole in the center were hung on ladder like rigs, as shown in Figure 15. The test site is in the bay outside the Sven Lovén Centre for Marine Sciences in Kristineberg, 100 km north of Gothenburg, Sweden. The rigs are removed annually from the water, specimens are cleaned of fouling (overgrowth) organisms, visually rated, and X-rayed. After this, the specimens are immediately placed at the sea bottom again. In this test, specimens with CAP as the matrix with an unmodified, an acetylated or a thermally modified wood component were included. Results show that the CAP unmodified wood WPCs were destroyed by shipworm attack after two years. Reference solid unmodified pine wood is often destroyed after one year. The CAP WPCs with modified wood are still sound after 6 years and the test is still in progress. Figure 16 shows an X-ray image of a sound board of CAP thermally modified wood WPCs after 6 years of exposure and one solid wood control board which was completely destroyed by shipworm after one year of exposure.
Figure 15. Test rig used for the marine test, left image: just after removal from the sea bottom at the annual evaluation, right image: after annual evaluation, before replacement at sea bottom.

Figure 16. X-ray image of an extruded CAP thermally modified wood WPC after 6 years exposure (left) and a solid wood control board after one year exposure (right).
5. MICROMORPHOLOGICAL FEATURES

5.1 CHANGES IN SIZE AND SHAPE OF THE WOOD COMPONENT DUE TO THE EXTRUSION PROCESSING

The thermoforming processing of WPCs requires both high temperatures (in general at least 170–180 °C) as well as intense shearing action in order to ensure sufficient mixing and dispersion of the wood component in the matrix. It is evident that such processing conditions may lead to severe degradation of the wood component, i.e. apart from the obvious thermal degradation, also a mechanical damage, and combinations thereof, with resulting change in size and shape (Rowell 2007). Studies by Glasser et al. (1999), Nitz et al. (2000) and Rowell (2006b) indicate that the extrusion of WPCs causes damage to the wood component, mainly as a reduction in size of the wood component. Gacitua et al. (2008) have applied a nanoindentation technique to investigate the damage to the wood cell wall caused by WPC processing. The processing in this case resulted in a reduction of the Young’s modulus of the S2 layer of the wood cell wall by 40-70%. Furthermore, latewood cell walls were found to suffer less collapse and damage than earlywood cell walls.

The damage done to the wood component varies depending on many parameters e.g. the inherent wood properties, the original size and shape of the wood component, the melt properties of the matrix, the effects of additives used, screw design and the size of the extruder. The effects of using different types of modified wood components on such extrusion-related mechanical degradation are investigated in Paper II. The wood components originated from solid acetylated, thermally modified and unmodified wood and were ground by means of a newly developed two-step process. This process enables a greater aspect ratio (fiber direction length vs. width) than conventional wood meal or sawdust raw material. The WPCs were extruded into hollow profiles on a conical extruder. Paper III involves unmodified and acetylated wood components which were injection molded into WPC tensile test bars. In both Papers II and III it was observed that the wood component became significantly reduced in size in all samples due to the thermoforming processing. The most
extensive changes could be observed for the thermally modified wood component. By isolating the wood component from the composites through an extraction process in boiling xylene, it was possible to analyze the wood component size and shape after processing (Paper II). Figure 17 shows the extracted wood components from the conical extruded WPCs with 70 wt.-% wood. The difference between the wood components can clearly be seen, i.e. the estimated average size of the thermally modified wood component is several times smaller than the acetylated and unmodified wood components. The shape of the acetylated wood component appeared to have a more splinter like shape than the unmodified wood component which had a more rounded shape.

Figure 17. Wood components extracted from the extruded WPCs with (a) thermally modified spruce, (b) acetylated pine, and (c) unmodified pine.

Micromorphological examination of the fracture surfaces revealed a higher amount of larger sized wood particles in the fracture zone for the WPCs with unmodified and acetylated wood components than in the WPCs with a thermally modified wood component. These larger wood components could possibly have less ability to redistribute stresses in the material than smaller ones, which could also explain why the WPCs with thermally modified wood had the highest tensile strength according to the micromechanical evaluation presented in Paper II.
5.2 SPECIMEN PREPARATION TECHNIQUE BY MEANS OF UV EXCIMER LASER ABLATION

There are several methods used for examining the inner micromorphology (or microstructure) of materials such as wood and composites. One way to study the microstructure of composites is to examine fracture surfaces after, e.g. tensile strength testing, from which the internal microstructure may be estimated. However, it must be emphasized that the microstructure in the fracture zone after such tests has most likely been deformed, and subsequently represents the weakest part of the material and not the average structure of the composite. Nevertheless, examinations of the fracture surfaces may provide important information, such as fiber-matrix compatibility, fiber embedment and fiber pullout.

A key challenge for the evaluation of the micromorphology of WPCs relates to the specimen preparation technique for preparing mechanically undamaged surfaces for scanning electron microscopy (SEM). Traditional techniques for preparing surfaces for microscopy of soft materials such as wood often involve an initial moistening followed by mechanical treatment of the surface, such as microtoming, razor blade cutting or polishing. Such techniques may lead to uncontrolled damage such as microcracks and distortion of the surface morphology as well as contamination and redistribution effects. It is especially difficult to prepare surfaces of material with two distinctively different phases such as in wood plastic composites.

A preparation technique based on UV excimer laser ablation is demonstrated in Paper I. This technique for ablating wood surfaces was developed at KTH (Stockholm, Sweden), in the early nineties (Stehr et al. 1998, Seltman 1995). The laser used is a pulsing UV excimer laser (LAMBDA PHYSIK LTD 210 ICC) that applies the wavelength 248 nm. Each pulse corresponds to 1 J/cm² energy and the pulse frequency was varied between 1 and 100 Hz in this study. Figure 18 shows the UV laser laboratory set-up and an SEM micrograph of a sawn wood cross section before and after ablation.
The technique has shown to be well suited for precision micromachining of wood and other lignocellulosics, as well as certain polymers and plastics. The prepared samples can reveal the micromorphology features of the composites, for example, the orientation and size of the wood component, cell wall damage, the collapse and compression of the cells, the polymer filling of the lumens, porosity and the interface delamination between the components.

5.3 MICROMORPHOLOGY OF THE WPC SAMPLES

Figures 19 to 22 in this section show SEM micrograph examples of the micromorphology of some WPC samples, where all surfaces have been prepared using the micromachining technique with UV excimer laser ablation. Figure 19 shows an injection molded WPC with 50 wt.-% unmodified wood and PP as the matrix. In this figure the following important micromorphological features can be seen:
Figure 19. Micromorphology of an injection molded WPC with 50 wt.-% unmodified wood (lighter colored phase) and PP as matrix (darker colored phase); a) cell wall fragments, b) earlywood tissue, c) compressed earlywood tissue, d) latewood tissue, e) interface delamination.

1) A pronounced variation in the size and shape of the wood component (lighter phase), i.e. from fine cell wall fragments (a) to larger portions of wood tissue or bundles of wood fibers (d). 2) The lumens of the wood tissue are filled with the thermoplastic matrix (darker colored phase), leading to a low porosity, normally in the range of a few percent, in the composites. 3) Wood tissue consisting of mostly latewood cells often shows no cell wall compression or collapse (d). 4) Wood tissue consisting of mostly earlywood cells often shows compressed or collapsed cells (b) or is separated into cell wall fragments (a). 5) Wood-polymer interfacial delamination or microcracks (e). The disintegration into cell wall fragments was mostly pronounced for the WPCs with a thermally modified wood component, which resulted in a more homogeneous WPC micromorphology than the WPCs with unmodified and acetylated wood.
components (Paper II). This was also reflected in the micromechanical evaluation where WPCs with a thermally modified wood component showed the greatest strength.

Figure 20 shows a micrograph of an injection molded WPC with a 50 wt.-% acetylated wood component. In the figure it can be seen that many of the wood particles have been split into single fibers and fiber fragments. No such extensive fragmentation could be observed in the WPCs with an unmodified wood component (Figure 19). This micromorphological observation is in good agreement with the light microscopy analysis of extracted wood component from the composites (cf. Figure 17).

![Figure 20. Micromorphology of an injection molded WPC with 50 wt.-% acetylated wood and PP as matrix.](image)

The local interfacial delamination as indicated in Figure 19, see the denotation (e) indicates a poor adhesion or compatibility between the hydrophilic wood and the hydrophobic PP matrix, and that such adhesion
properties may vary greatly within the composites. In a parallel investigation to this thesis (Bryne and Wålinder 2010 and Bryne et al. 2010), surface energy and surface chemical composition characteristics of modified wood and so-called wood-thermoplastic interaction or adhesion parameters have been studied; and the results indicate that acetylated wood has distinctly different adhesion abilities with certain thermoplastics. Another possibility to study such wood-polymer adhesion and to explore this area further would be by using a technique called inverse gas chromatography (IGC), see e.g. Tze et al. (2006). This technique is suggested as a possible future area of research related to this thesis.

When WPCs are processed, moisture is ventilated out from the composite so the final product contains very little moisture, less than the equilibrium moisture content of the wood component in most environments. This implies that the WPC gains moisture with time and the wood component swells due to the moisture uptake. Depending on the level and rate of uptake as well as cyclic moisture gain and loss, irreversible deformations of the WPC may occur along with interfacial delamination between the wood and thermoplastic. The micromorphology of extruded WPCs with 70 wt.-% wood and PP is evaluated before and after moisture soaking and drying in Paper V. Figure 21 shows a micrograph of a WPC with unmodified wood subjected to one cycle of water soaking followed by drying. There large cracks are visible between most of the wood components and the thermoplastic matrix. The WPCs with acetylated and thermally modified wood components were also subjected to the same soaking procedure, but crack formation at wood-polymer interfaces was greatly reduced due to the more dimensionally stable wood component (Figure 22).
Figure 21. Extruded PP-unmodified wood WPC with 70 wt.-% wood after one soaking drying cycle.

Figure 22. Extruded PP-acetylated wood WPC with 70 wt.-% wood after one soaking drying cycle.
6. CONCLUSION AND FUTURE WORK

6.1 CONCLUSIONS

The work presented in this thesis demonstrates the feasibility of using a modified wood component in wood plastic composites. The incorporation of a modified wood component into WPCs allows for a higher wood content without compromising on the long term durability of this type of material. The following conclusions can be drawn from this thesis:

- The use of a modified wood component in WPCs decreases moisture movements in the material. Accelerated weathering or the use of a hygroscopic matrix material will increase the rate of moisture sorption into the composite, however the final moisture levels are unchanged.
- WPCs with an acetylated wood component showed greatly reduced crack formation caused by moisture induced movements than compared to WPCs with an unmodified wood component.
- Laboratory decay tests show low mass losses due to decay for WPC materials. However, a mechanical evaluation prior to and after testing may reveal losses in strength due to decay at an earlier stage than mass loss.
- High wood content WPCs with a modified wood component perform well in field testing. After a 7 year in ground test all composites, except the high wood content WPCs with an unmodified wood component, are considered sound.
- Mechanical action during processing severely damages the wood component. The damage was most pronounced for the thermally modified wood component, which after processing showed significantly shorter and more damaged wood tissues than before the extrusion process.
- Surface preparation by means of an UV-excimer laser ablation technique was shown to be well suited for analysis of the micromorphology of these types of wood-plastic combinations.
6.2 FUTURE WORK

Future challenges for WPCs are, among others, to try to tailor interface properties in order to achieve a composite that performs well even after it has been subjected to moisture and temperature fluctuations. By analyzing the surface properties of the different wood components as pertains to surface energy, wetting properties and surface heterogeneity, and comparing these to different thermoplastics properties, theoretical predictions of interaction parameters can be obtained for different wood thermoplastic combinations. Inverse gas chromatography (IGC) will be used to study surface energetics (gas-solid interactions) and surface heterogeneity of the components. This will give a basis for the theoretic determination of the properties for different wood-thermoplastic combinations. Such measurements, in combination with practical adhesion studies, may contribute to the knowledge about adhesion phenomena in wood plastic composites (WPCs).
7. REFERENCES


APPENDED PAPERS (I-VII)

Division of the work in the appended papers:

I. Wålinder, M.E.P., Omidvar, A., Seltman, J. and Segerholm, B.K. “Micromorphological studies of modified wood using a surface preparation technique based on ultraviolet laser ablation”

Wålinder initiated the work. Segerholm, Omidvar and Seltman performed the experiments. Segerholm and Wålinder interpreted the results and jointly wrote the paper.

II. Segerholm, B.K., Vellekoop, S. and Wålinder, M.E.P. “Process-related mechanical degradation of the wood component in high-wood-content wood-plastic composites”

Segerholm and Wålinder initiated the work. Segerholm and Vellekoop performed the experiments and all authors jointly interpreted the results. Segerholm wrote the paper.

III. Segerholm, B.K., Walkenström, P., Nyström, B., Wålinder, M.E.P. and Larsson Brelid, P. “Micromorphology, moisture sorption and mechanical properties of a biocomposite based on acetylated wood particles and cellulose ester”

The consortium of Ecocomp initiated the work. Segerholm performed the sorption and micromorphological experiments, Walkenström performed the mechanical characterisation, Nyström performed the particle characterisation. The authors jointly interpreted the results. Segerholm wrote most of the paper.
IV. Segerholm, B.K., Ibach, R.E. and Wålinder, M.E.P.
“Moisture sorption in artificially aged wood-plastic composites”

Segerholm and Ibach initiated the work, performed the experiments and all authors jointly interpreted the results. Segerholm wrote the paper.

“Improved Durability and Moisture Sorption Characteristics of Extruded WPCs made from Chemically Modified Wood”

Segerholm initiated the work. Segerholm performed the sorption experiments. Westin and Alfredsen performed the durability tests and all authors jointly interpreted the results. Segerholm wrote the paper.

VI. Segerholm, B. K., Ibach, R. E., and Westin, M.
“Moisture sorption, biological durability, and mechanical performance of WPC containing modified wood and polylactates”

Segerholm initiated the work. Segerholm, Ibach and Westin performed the experiments. All authors jointly interpreted the results. Segerholm wrote the paper.

VII. Segerholm, B. K., Larsson Brelid, P., Wålinder, M. E. P., Westin, M., and Frisk, O.
“Biological outdoor durability of WPC with chemically modified wood”

Westin initiated the work. Westin and Larsson Brelid performed the durability assessment. All authors jointly interpreted the results. Segerholm and Larsson Brelid jointly wrote the paper.