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Section 4 **Processes and Properties**

WOOD PLASTIC COMPOSITES FROM MODIFIED WOOD
Part 3. Durability of WPCs with bioderived matrix

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WOOD PLASTIC COMPOSITES FROM MODIFIED WOOD
Part 3. Durability in laboratory decay tests

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ABSTRACT

The decay resistance of fully bio-derived wood plastic composites, WPCs, was tested in both laboratory and field tests. The laboratory tests were performed according to modified versions of AWPA E10 (soil-block test) and ENV 807 (tests in three un-sterile soils) and the field tests according to EN 252 (stakes in ground) and EN 275 (resistance to marine borers). The WPC materials for laboratory tests were injection molded test specimens with 50% modified wood particles and 50% cellulose ester (CAP) or poly-lactic acid (PLA) content. The field test specimens were taken from larger extruded decking board profiles with 60% wood content and 40% CAP. 60/40-mix (wt/wt) for CAP corresponds to the same volumetric composition as 70/30-mix (wt/wt) with polypropylene as matrix that was presented in Part 1 and 2.

In all laboratory tests the control WPCs performed much better than the pine sapwood control blocks. The WPCs from modified wood performed better than the control WPC and WPCs from acetylated wood performed best with no detectable decay whatsoever.

In the field stake test, the WPC from unmodified wood were slightly decayed whereas the WPCs from modified wood were sound. In the marine field test the WPC from unmodified wood were severely attacked by shipworm (*Teredo navalis*), whereas the WPCs from modified wood were sound.

Keywords: Durability, WPC, Wood Plastic Composite, Modified wood, Bio-plastics, Cellulose ester, PLA.

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1. INTRODUCTION

Wood plastic composites (WPCs) have been marketed as highly durable and maintenance-free, since they were introduced on the American market. However, the first generation of WPCs without preservatives (e.g. zink borate), UV absorbers and coupling agents did not fulfill the expectations of the customers – there has been several cases of severe decay in-service which often led to so called “class action law suites” (Morris and Cooper 1998). The second generation, with the above mentioned additives, is initially performing better, although laboratory testing indicates that they still may fail under certain conditions in-service because of their high content of unmodified non-durable wood particles (Ibach et al. 2003, Ibach and Clemmons 2006).

An alternative approach to achieve durable WPCs is to use modified wood particles which will be more compatible with the plastic matrix and more durable, which may possibly make it unnecessary to add coupling agents and preservatives (Ibach and Clemmons 2003 and 2006, Westin et al. 2006).

In the two first parts of the project presented at the meeting in Tromsö (Larsson-Brelid et al 2006, Westin et al 2006), results for WPCs from modified wood and polypropylene a petroleum derived polymer, were presented.

In this part, the durability of fully bio-derived WPC materials made from modified wood and bioderived polymers, is assessed.

2. MATERIALS AND METHODS

2.1 Wood material

The wood material used was sapwood of Scots pine (*Pinus sylvestris*) for acetylation and reference treatments, Norway spruce (*Picea abies*) for thermal modification and radiata pine (*Pinus radiata*) for furfurylation. All Scots pine sapwood came from Unnareds sawmill in south west of Sweden. The Norway spruce came from a StoraEnso sawmill in Finland and the radiata came from Spain.

The acetylation (with acetic anhydride) was performed on sapwood boards of dimensions 24 x 125 x 2000 mm^3. All the boards were first kiln-dried to a moisture content (MC) of approximately 5 percent. The boards were then acetylated in SP’s pilot plant according to the simplified procedure further described in Rowell et al. (1986) and Larsson Brelid (1998). The acetic anhydride was purchased from BP Chemicals and was of a technical grade (concentration >97.9%). The average degree of acetylation was approximately 21% expressed as wood acetyl content, calculated from the liberated amount of acetate determined by HPLC after de-acetylation of oven-dried wood particles with 1M sodium hydroxide (Paulsson *et al.*, 1994). The furfurylation was performed in the production plant of Kebony Products ASA (now Kebony ASA) using an impregnation solution with an FA concentration of 27% and their normal production process at the time. The resulting WPG was approximately 30%. The Norway spruce was treated according to ThermoWood D (212°C peak temperature) process by StoraEnso in one of their ThermoWood process plants. As references, Scots pine sapwood samples were treated with CCA (Chromated copper arsenate) preservative to two CCA retention levels using 1.3 and 0.6% CCA concentration, respectively.

The preparation of the wood particles was carried out according to a two-step process. In the first step, the modified and unmodified solid wood boards were cut into 24 x 125 x 190 mm^3 blocks which were fed into a disk flaker (Bezner) and processed into thin veneer strands of the type used for the production of oriented strand board (OSB). In the second step, the veneer strands were fed into a dry grinding knife-mill (Condux) and chopped into fine particles. The principal idea in this work is to use residuals from modified solid wood production, which is the reason for first modifying solid wood boards and then grinding them into particles.

2.2 WPC material

The matrix used for most of the WPCs presented in this paper, was cellulose acetate propionate, CAP (Tenite CAP 360A4000016, Eastman Chemical Company, ρ = 1190kg/m^3, melt flow rate: not available). The CAP product contained 16% plasticizer, DOA (bis(2-ethylhexyl) adipate). The high plasticizer content increases the processability by decreasing the viscosity and thereby facilitates the incorporation of a high content of wood particles. For some of the WPCs, a commercial grade of PLA (poly-lactic acid) from NatureWorks, 4042D, was used.

Injection molded wood-CAP-composite samples:
Prior to compounding, the wood particles were dried in an oven at 105ºC for 4 hours. The constituents were compounded in a Werner & Pfleiderer ZSK 25 WLE co-rotating twin screw extruder with a length-to-diameter ratio of 44. In this work, the wood particle content in the composite was 50% by weight. Two individually controlled gravimetric K-tron feeders from Christian Berner were used for particle weight fraction control. The barrel temperatures was 185-220 ºC and the screw speed was 150 rpm. The compound was pulled through a water bath and cut to granules 4 mm long and approximately 4 mm in diameter using an SF Sheer granulator. The granules were oven-dried at 75ºC for three hours followed by injection molding in an Engel ES 200/110HL machine into dumbbell-shaped tensile test bars ("dog-bones").

Injection molded wood-PLA-composite samples:
Furfurylated wood particles and untreated wood particles were mixed 50 wt% into PLA by extrusion compounding using a Berstorff ZE40-38D twin screw extruder (see Figure 1). These compounds were injection molded into flexural test bars.

Figure 1: Extrusion compounding facility at A&F used for compounding of furfurylated wood particle-PLA WPC material.

Extruded decking profiles:

The compounding was carried out as full scale experiments at the company OFK Plast in their counter-rotating twin screw extruder.

Figure 2: Collecting granulates of the WPC compound at OFK Plast.

Figure 3: Examples of different types of compounds produced

Each compound was used for production of several meters of decking boards at OFK Plast, and for production of a few meters of hollow window frame profiles at Primo AB.

Figure 4: Manufacturing of WPC decking boards at OFK Plast.

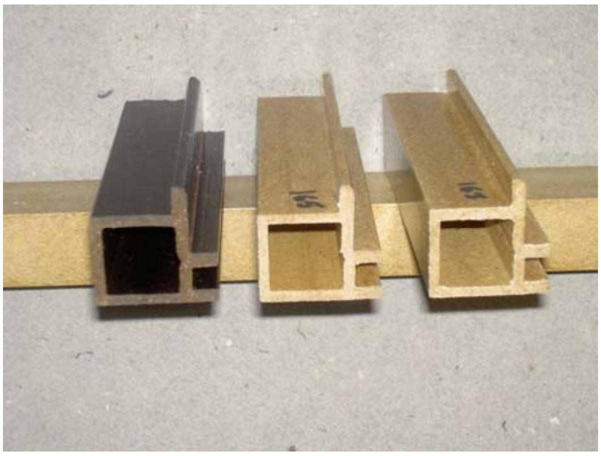

Figure 5: Extruded window profiles (left to right: WPC profiles from furfurylated, acetylated and untreated particles, respectively)

Processing problems occurred in the manufacturing of decking boards from the furfurylated RP/CAP compound. However, at Primo the furfurylated/CAP was easily processed the window profiles. The decking boards were used for all decay tests except for laboratory tests of WPCs from furfurylated wood for which the window profiles were used.

2.3 Pre-ageing of laboratory test specimens, mechanical and physical testing

Pre-ageing

All test specimens were leached according to EN84, i.e. leaching two weeks in de-ionized water (5:1 water to test sample volume ratio) with water exchange 10 times and then oven-dried 18h. The injection molded CAP-WPC specimens were exposed for 1000h (500h on each flat side) in a weatherometer. All WPC specimens were soaked 14 days in de-ionized water. The ENV 807 specimens were directly inserted in the test soils after the water soak. The E10 specimens were sterilized by gamma-radiation.

Preparation of specimens by UV laser for LV-SEM

For the specimen preparation, 10 x 10 x 4 mm^3, blocks were cut from the waist of the injection moulded dumbbell-shaped tensile test bars. From these blocks, rectangles with dimensions of 10 x 4 x 1.5 mm^3 were detached by UV laser. The preparation technique involving UV laser cutting of surfaces leaves the interior of the material unaffected. To determine the micro-structure of the WPCs, the thin UV laser cut specimens were studied, using a scanning electron microscope (SEM), *JSM-5310LV* (Jeol Ltd., Tokyo, Japan). The microscope was run in a low vacuum mode (LV-SEM) without need for gold-sputtering the surfaces of the specimens.

Mechanical tests

Tensile tests (ISO 527-1, 5 mm/minute and 1 mm/minute for modulus) were performed on the tensile test bars, using MTS 20M equipment (MTS Systems Corporation). The modulus of elasticity was calculated from the stress-strain curves between strain values of 0.05-0.25%. Flexural properties were measured on a Zwick 1445 universal testing machine according to ISO 178 at a support length of 64 mm. The crosshead speed was 2 mm/min for the modulus and 10 mm/min for the strength. Flexural properties were determined from 5 specimens per batch. Charpy unnotched impact strength was determined using a Ceast pendulum impact tester according to ISO 179 using an impact hammer of 4J at a speed of 2.9 m/s. The Charpy impact strength was determined from 8 specimens per batch. HDT (Heat Deflection Temperature) of injection moulded specimens before and after annealing at 105°C was evaluated at 1.35 MPa load, at a strain of 0.2% and at heating rate of 2°C/min.

Water vapor sorption tests

Prior to the sorption test, half of the test bars from each type of WPC and also pure CAP and PP were subjected to accelerated ageing in an Atlas Weather-Ometer® 65% (WoM) with a 6500 W xenon arc lamp with spectral irradiance 0.35 W/m^2/nm at 340 nm. The WoM was programmed for 102 minutes of water spray followed by 18 minutes of light and water spray according to ISO 11 341:1994, method 1, wetting cycle A. Total exposure time in the WoM was 500h on each side. The specimens used were the original injection molded test bars with the flanges cut of. Specimen dimensions were 170x10x4 mm^3, specimen holders on top and bottom of specimens, leaving 150 mm exposed surface in the middle. The other half of the test bars, together with acetylated and unmodified solid wood, was tested without any pre-ageing. Two replicates of each test material were cut and planed into specimens with the dimensions 24 x 10 x 2 mm^3 leaving one (24 x 10 mm^2) original outer surface of the injection molded specimen unaltered. All the other surfaces were then sealed with aluminum tape and placed in a desiccator with silica gel for 4 weeks prior to the test. The test was carried out by placing the specimens in an enclosed chamber above a saturated ammonium chloride solution, giving the ambient air a relative humidity of approximately 80% at 22 °C. The specimens were weighed at certain time intervals until the equilibrium moisture content (EMC) was reached, and the climate was recorded by a temperature and humidity logger placed inside the chamber. The specimens were dried after reaching the EMC in order to determine the initial and final MC.

2.4 Laboratory decay tests

Soil-block test (AWPA E10)

The soil-jars were steam-sterilized twice, first time with only the soil and second time with Scots pine feeder strips on the soil. One WPC test specimen (5x15x30mm in first set and 5x10x30mm in the second set) and one pine control specimen were put on the feeder strips of each soil-jar, which was then inoculated with mycelium suspension of respective test fungi according to Segaye et al. (1996). The duration of the test was 2+8 weeks (2 weeks for the initial over-growth and 8 weeks for the actual test). The brown rot test fungi were *Postia* *placenta* (Fries) Cooke sensu J. Eriksson (FPRL 250), *Coniophora puteana* (Schumacher ex Fries) Karsten (BAM Ebw. 15) and *Gloeophyllum trabeum* (Persoon ex Fries) Murrill (BAM Ebw. 109).

TMC test (according to ENV 807)

The 5x10x100mm-specimens were buried to ¾ of their length in the soil in three types of terrestrial microcosms, TMCs, according to an expanded version of European test standard ENV 807. The soils were: a compost soil (TMC 1), soil from the Simlångsdalen test field (TMC 2) and soil from a mixed forest (TMC 3). Specimens were removed after 32 or 40 weeks (except for tests of PLA-WPCs which were removed after 47 weeks) after which the mass loss was calculated.

Figure 6: Test set-up for TMC test (ENV 807), illustrated by the soil box with soil from the old Simlångsdalen test field.

The compost soils was a mixture of 2/3 "Thomas Nilsson No 1" with high activity of both tunneling bacteria and softrot and 1/3 of Borås municipal compost from a mix of household and garden waste. The compost soil mix had a pH of 7.4 and a Water Holding Capacity (WHC) of 102%. The test soil from the test field in Simlångsdalen is a sandy soil with dominating brown rot decay (mainly caused by *Leucogyrophana pinastri*), pH=5.2 and WHC=20%. The forest soil was from the test field in Ingvallsbenning (with high activity of the white rot fungus: *Asterostroma cervicolor*). The forest soil had pH=4.6 and WHC=130%.

2.3 Field tests

In-ground stake test (EN 252)

The test stakes were put in the field in SP's test field in Borås early June 2005. The test field has active brown, white rot and soft rot. Soft and white rot decay types are dominating with streaks of brown rot across the field. The annual precipitation is slightly below 1000 mm. The test stakes are assessed annually.

Marine borer resistance test (EN 275)

The test rigs are placed in the bay outside Kristineberg Marine Research Station, 100 km north of Gothenburg, Sweden.

Borer activity (the characteristics listed are for the actual test site): The gribble *Limnoria lignorum* (Rathke) present but not very active at the test depth; *Teredo navalis* L. (most occurring mollusc specie) up to 7cm length during one growth season; *Nototeredo norvagica (Spengler)*, up to 15 cm length during one growth season. There is borer activity all around the year. The most common borer specie, *Teredo navalis*, is active 11 months out of 12. The criterium for failure due to teredinid attack (tunnels covering more than 50% of the specimen area as it appears on the X-ray film) is often fulfilled already within 6 months for untreated control specimens. Maximum "service" life of untreated controls is approx. 1.5 years, after which the samples are often broken to pieces by the autumn storms.

Specimens were hung on nylon-bars (Ø=24mm) of two ladder-like rigs placed on the sea-bottom at 6±0.4 m depth at low tide. The test rigs have heavy concrete foundations and each rig was kept upright by a 50cm-diameter buoy. As a result of the arrangement, the specimens are located at depths varying from 4 m to 6.5 m below water surface during the exposure period. The test was started in 2006.

Annually, the rigs are taken up, the tests specimens cleaned from fouling (overgrowth) organisms, rated visually for possible attack by *Limnoria*, X-rayed and immediately put back after this was ready. The X-ray films were developed and the teredinid attack rated after inspecting the X-ray films on a light-table. The wood samples that had been given the rating: failure, were rejected and the remaining samples were put back on the rigs which were then put back into the sea.

3. RESULTS AND DISCUSSION

3.1 Mechanical properties of the WPCs

CAP-WPCs

Results from tensile tests of injection molded dog-bones show that the tensile strength of CAP-WPCs generally is better than for the PP-WPCs (presented in Part 1). The tensile strength for control WPCs (50% unmodified wood, 50% CAP) was approx 30 MPa and the strength was better for WPCs with modified wood particles, especially heat treated (37 MPa) and acetylated (43 MPa). The strain and modulus of elasticity (MOE) was also higher (>6 GPa) with modified wood particles. However, the fracture energy was slightly reduced.

PLA-WPCs

Processing of both untreated and furfurylated wood particles in PLA exhibited foaming. Nevertheless, suitable materials for further evaluation could be obtained. However, injection molding into test bars with no irregularities was possible.

Both the untreated and furfurylated wood particle-PLA composites showed higher flexural stiffness, lower flexural strength and impact strength than pure PLA (Table 1 & 2). Variation in data was small. The mechanical properties of the furfurylated wood-PLA composites are in the same range as those of commercially available WPC materials, see reference value indicated in Table 1 and 2.

The heat deflection temperature (HDT), a measure for the maximum temperature at which a material can be reasonably applied, is also of importance for PLA based WPC materials, since PLA has a glass transition temperature (Tg) of circa 60°C and the material weakens around this temperature. This temperature may be reached when serving as decking in full summer. The HDT value of PP, PE and PVC based WPCs usually increases upon incorporation of reinforcing fibers. The HDT of the PLA composites however did not improve upon addition of wood fibers (Table 2). Increasing the PLA's degree of crystallinity by annealing at 105°C for a couple of hours, however, also resulted in increased HDT-values for the PLA composites of over 100°C. This shuld be sufficient for service in decking and siding applications. For the furfurylated wood WPC samples, a nearly amorphous PLA grade was used as matrix by mistake, therefore the HDT of this particular composition did not improve significantly upon annealing.

Table 1: Mechanical properties of PLA compounds and commercial WPC materials.

	Stiffness		Bending Strength		Strain at max. S.	
	[GPa]	std	[MPa]	Std	[%]	std
PLA, injection molded	3.1	0.1	107	1	4.7	0.1
PLA, extruded	3.1	0.1	108	1	4.8	0.1
50% Untreated wood in PLA	5.4	0.1	89	2	1.9	0.1
50% Furfurylated wood in PLA *	7.0	0.2	87	3	1.3	0.1
70% Softwood in PP (commercial Techwood) [4]	*7.2*		*73*			

Table 2: Mechanical and thermal properties of PLA compounds and commercial WPC materials.

	Charpy Impact		HDT	HDT after 4h at 105°C
	[kJ/m^2]	std	[°C]	[°C]
PLA, injection molded	23.8	3.5	51	61
PLA, extruded	27.1	5.4	50	61
50% Untreated wood in PLA	8.3	1.2	52	121
50% Furfurylated wood in PLA *	5.2	0.7	50	61*
70% Softwood in PP (commercial Techwood) [4]	*7.6*			

* An amorphous PLA grade, NatureWorks 4060, was used by mistake.

3.2 Physical properties of the WPCs

CAP-WPCs

Water vapour sorption data for the composites are shown in Figures 7 and 8. The composites with CAP matrix picks up moisture more rapidly than the composites with PP as matrix (presented in Part 1: Larsson-Brelid et al. 2006), since the CAP matrix is more hygroscopic than the PP matrix.

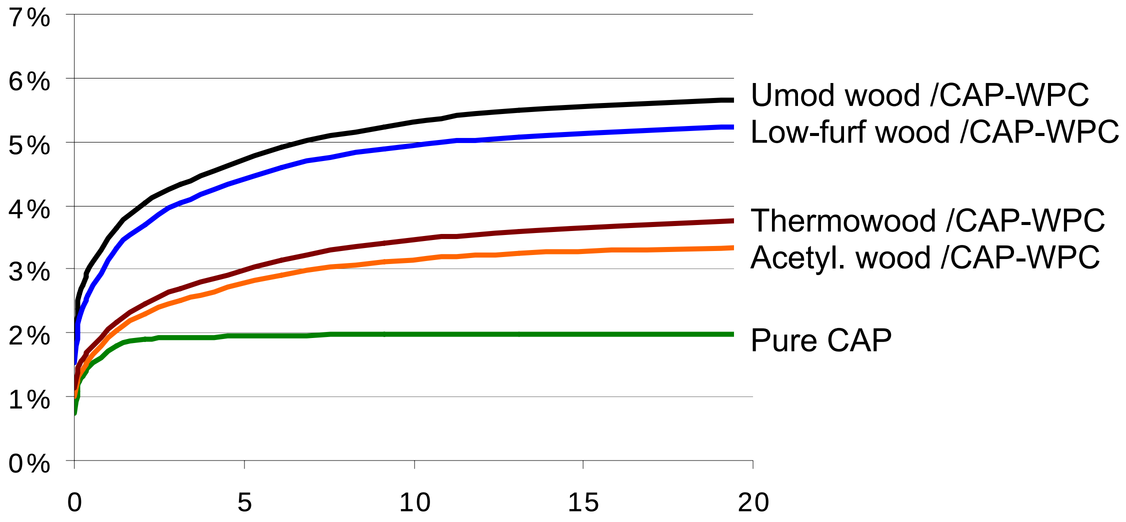

Figure 7: Moisture sorption curves of thin slices of WPC with CAP as matrix. The samples were aged in weatherometer 1000h before test.

The EMC seemed to be reached after 40 days (=> 1850 $s^{½}$) in the composites with CAP and unmodified wood, and after 30 days (=> 1600 $s^{½}$) in the composites with CAP and acetylated wood. However, true EMC was reached in half a year. The pure CAP sample reached EMC more rapidly, after about 5 days. There was no difference in time to reach EMC between non-aged and aged samples with CAP as matrix. In contrast, there is a significant difference between non-aged and aged samples with PP as matrix. The non-aged PP-composites did not reach equilibrium even after 500 days of sorption.

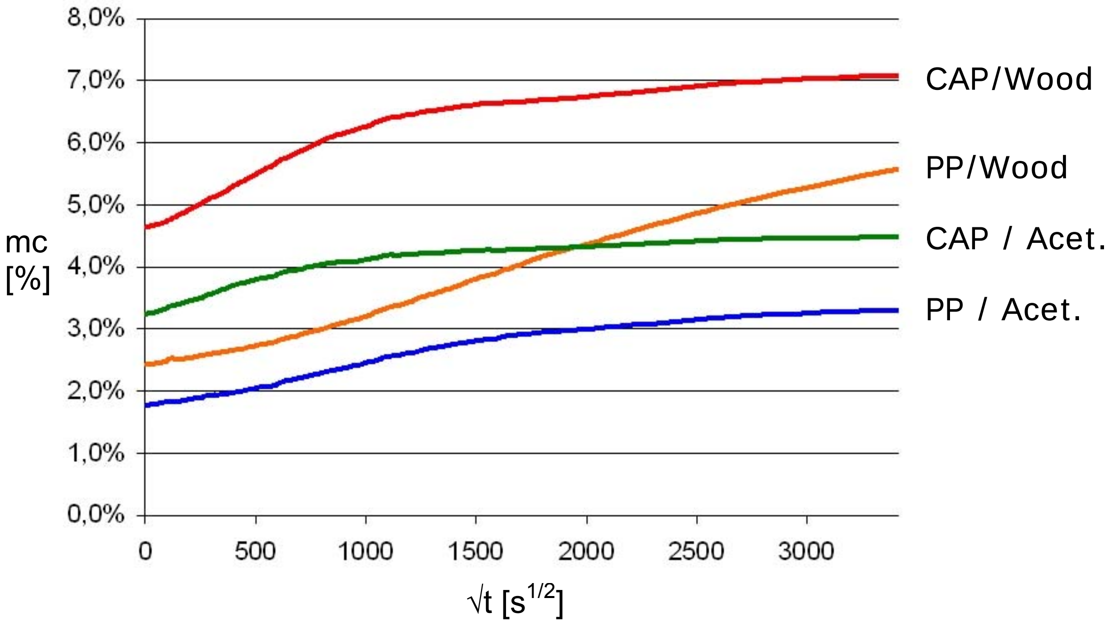

Figure 8: Moisture sorption curves of thin slices of WPC with CAP as matrix during longer test duration compared with WPCs with PP-matrix. The samples were aged in weatherometer 1000h before test.

The measurements also show large differences in EMC of the samples. After exposure, specimens based on acetylated wood and CAP had a final moisture content of 4%, in both aged and non-aged specimens. Specimens with unmodified wood and CAP reached a final moisture content of about 7% in both aged and non-aged specimens. The final moisture content of the pure CAP sample was 2%. Similar differences between the unmodified and acetylated wood components were also evident for the composites containing PP as matrix.

The final MC was equal to the EMC in all cases except for the non-aged composites based on PP, which did not reach their EMC. The EMC of the wood component was about 12% in all the composites containing unmodified wood. In the composites based on acetylated wood particles, the EMC was only 6%, so that acetylation of the wood reduced the EMC by 50%. These results suggest a strong reduction in the swelling and shrinkage of the wood component in the composites with acetylated wood. A reduced swelling and shrinkage will also reduce the interfacial stresses between the matrix and the acetylated wood subjected to a moist environment compared with those in unmodified wood. The lower moisture level associated with the modified wood component is also expected to increase the biological resistance of the composite.

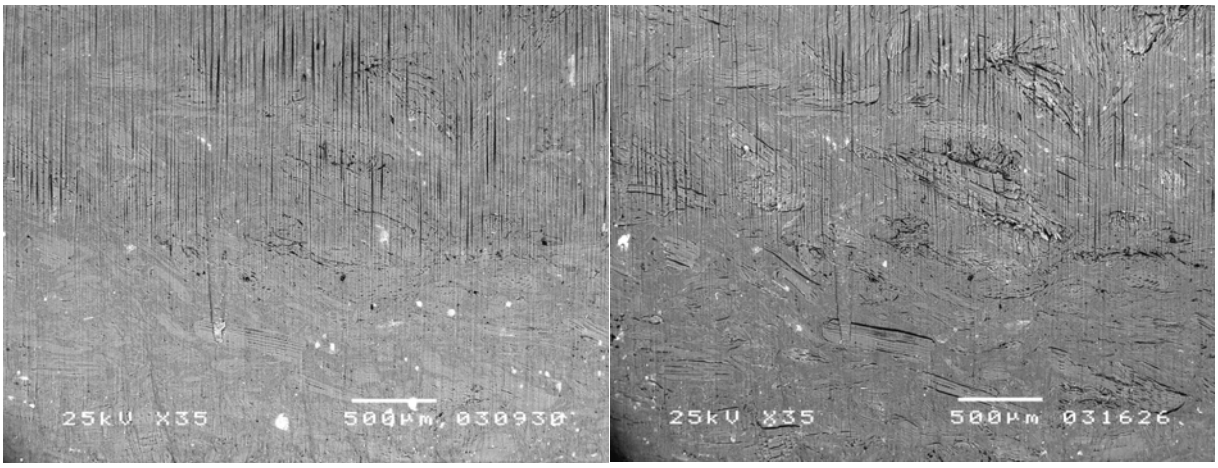

Figure 9: LV-SEM micrograph of control WPC (unmodified wood/CAP) before and after one water soaking/drying cycle. Center part of cross section of decking board.

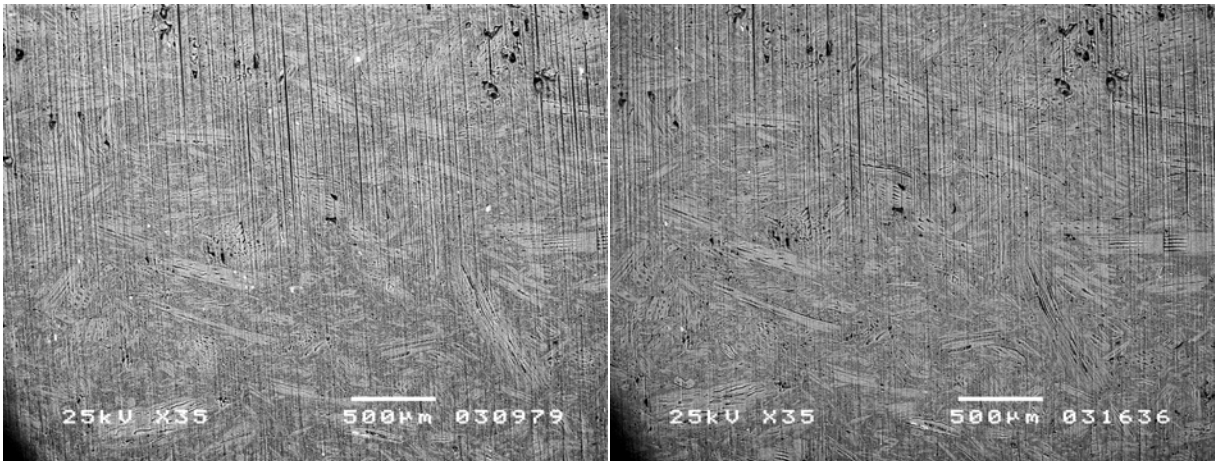

Figure 10: LV-SEM micrograph of acetylated WPC (acetylated wood/CAP) before and after one water soaking/drying cycle. Center part of cross section of decking board. The straight vertical stripes in the upper half of the pictures are artifacts from the laser-cutting.

UV laser prepared surfaces of the composites are shown in Figures 9 and 10. Micrograph 9b (after water soaking cycle) shows that a lot of interfacial cracks are formed around the particles of unmodified wood, contrary to the situation for acetylated wood particles shown in 10b. Close-ups reveal also internal micro-cracks for the unmodified wood where the cracks go not only through the radial rays, which could be a weak point, but also through the middle lamella of the wood particles. Internal cracks in the particles can be critical when the composite is loaded mechanically. Such cracks create additional stresses in already stress-concentrated regions at the interface between the particles and the matrix, and this will lower the mechanical performance of the composite.

3.3 Soil-block test (AWPA E10)

The results from the soil-block test shown in table 3 indicates good virulence of the *Poria placenta* and *Gloeophyllum trabeum* test fungi, i.e. mass loss values of 30% and above for pine sapwood controls. Furthermore, the virulence (13.3% ML) for the white rot fungus *Trametes versicolor* is acceptable considering the fact that the duration time was shorter than the standard prescribes for white rot. However, the virulence of *Coniophora puteana* was rather poor (14.8% ML of pine controls).

The mass loss due to fungal decay was very low for all WPCs with CAP matrix. There was some very slight detectable decay for WPC Controls (WPCs from unmodified wood) tested with *Poria placenta* and possibly some even more slight decay with *Coniophora puteana*. For the WPCs with modified wood, no decay could be detected in any sample and the mass loss values were close to zero.

Table 3: Mass loss (%) values for injection molded WPC specimens in soil-block test (AWPA E10).

	Corrected Mass Loss (ML) values in soil-block test with			
Material	***Postia placenta* (%)**	***Coniophora puteana* (%)**	***Gloeophyllum trabeum* (%)**	***Trametes versicolor* (%)**
Pine sapwood control	30,0	14,8	35,3	13,3
Inj.m. WPCs, 50% CAP				
50% Unmod. pine	3,1	1,3	0,5	0,5
50% Furfuryl. radiata	0,0	1,0	0,0	0,0
50% ThermoWood D	0,1	0,5	n.a.	0,0

* uncorrected values

3.4 TMC test (ENV 807)

The results shown in Table 4 are from the three soil-boxes. The mass loss (ML) values for the pine controls were 50% in the compost soil, 47% in the Simlångsdalen soil and 16% in the forest soil.

Table 4: Mass loss values for injection molded WPC specimens after 40 weeks exposure in the different soils.

Material	Mass loss (%) in Compost soil	Mass loss (%) in Simlångsdalen soil	Mass loss (%) in Forest soil
Pine sapwood specimens			
Pine sapwood control	50,2*	46,6*	15,5*
CCA ref. treated, HC3	11,1*	2,7*	4,1*
CCA ref. treated, HC4	5,5*	0,9*	3,0*
Pure CAP	1,8*	0,9*	0,4*
Inj.m. WPCs, 50% CAP			
50% Unmod. pine	9,2	6,0	5,5
50% Furfuryl. radiata	4,4	0,0	2,1
50% ThermoWood D	0,8	0,1	0,7
50% Acetylated pine	0,0	0,8	0,8

* uncorrected values

The ML for the CCA-reference treated pine was low at the 9 kg/m³ retention level (which is the requirement level for the Nordic Wood Preservation Council Class A approval – which means for use in Hazard Class 4), except in the compost soil (5.5% ML). There was more or less no ML for the pure CAP reference specimens in any of the soils. The corrected ML of the

WPC controls was 9.2% in the compost soil, 6% in the Simlångsdalen soil and 5.5% in the forest soil, based on dry composite mass. Microscope analysis showed that there was indeed fungal decay in all soil types for the control WPC. The ML for the WPCs from fyrfurylated pine was much lower –4.4% in the compost soil, 2.1% in the forest soil and no ML in the Simlångsdalen soil. Finally, there was very low ML for the WPCs from acetylated pine or heat treated spruce and no fungal decay could be detected in any of the specimens.

Table 5 show the ML values for test specimens sawn from extruded decking boards after 32 weeks exposure in the the three soils. The ML for the pine controls were 33.1% in the compost soil, 50.7% in the Simlångsdalen soil and 26.8% in the forest soil. The ML values for CCA reference treated pine specimens from another trial with very similar ML values for controls were included (and were quite similar to the values reported in table 4). The performance of the specimens from extruded WPC was similar to the performance of the injection molded WPC specimens (approx. the same x-values). There was slight decay in the control WPCs but no detectable decay in the WPCs from acetylated or heat treated wood.

Table 5: Mass losses (%) for extruded WPC specimens after 32 weeks exposure in the different soils.

Material	Mass loss (%) in Compost soil	Mass loss (%) in Simlångsdalen soil	Mass loss (%) in Forest soil
Pine sapwood specimens			
Pine sapwood control	33,1	50,7	26,8*
CCA ref. treated, HC3	3,6**	5,1**	5,7**
CCA ref. treated, HC4	0,7**	1,3**	4,7**
Extr. WPCs, 40% CAP			
60% Unmod. pine	6,1	4,0	7,7
60% Furfuryl. radiata	2,5	0,0	2,7
60% ThermoWood D	0,3	0,3	0,7
60% Acetylated pine	0,3	0,2	0,6

* uncorrected values, ** from another test with approx the same mass loss values

PLA-WPCs

In figure 11, the mass loss of specimens of WPCs with PLA as matrix is shown with pure PLA specimens as reference. The soil exposure period was in this trial almost 1 year and unfortunately no pine sapwood controls were included. Furfurylation of wood significantly reduces decay illustrated by 4-5% ML compared to up to 43% ML for the control WPC. Unfilled PLA did not have any mass loss at all.

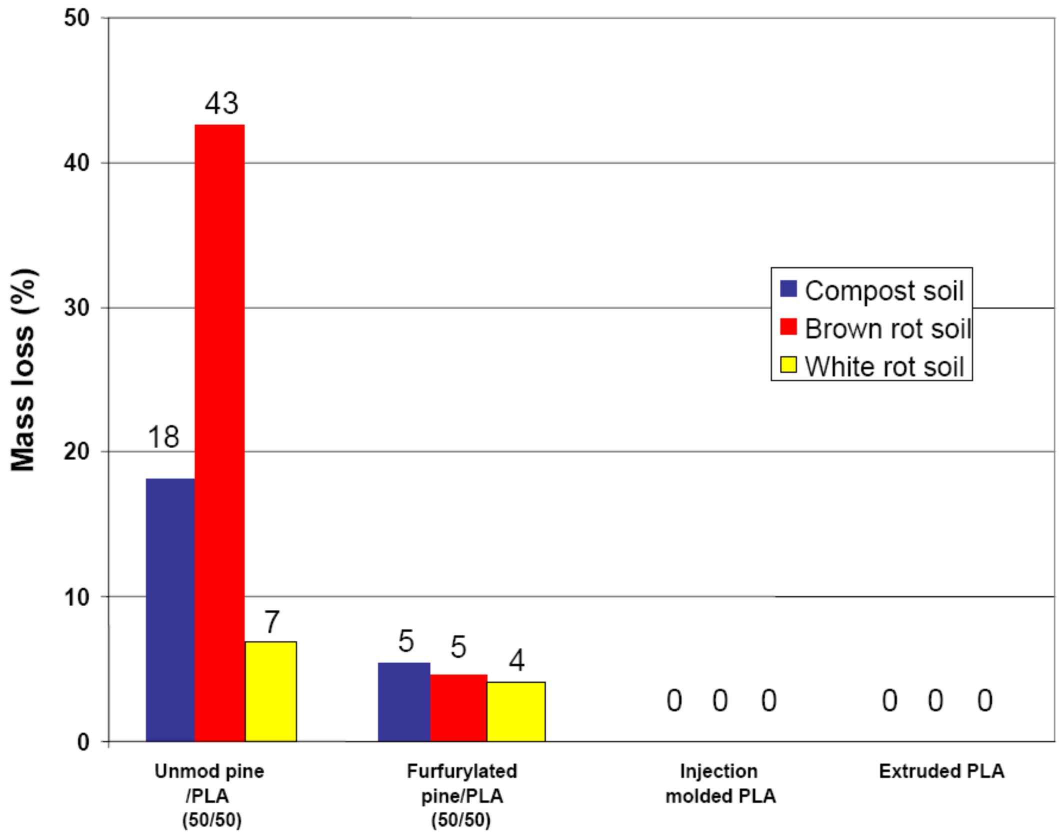

Figure 11: Results after 47 weeks in TMC test of PLA-WPCs.

The mechanical properties after soil burial tests are presented in Tables 6 and 7. Whereas the mass loss is limited to 5% for the furfurylated wood/PLA WPCs and 7-43% for the control WPCs, reduction in mechanical properties is substantial. Stiffness decreases by a factor of 2, flexural strength and impact strength reduce by a factor of 8 for the furfurylated wood particle composites. The control WPCs (with untreated wood particles) were so weak after the EN 807 test that they could not even be tested for mechanical properties (they fell to pieces when handled).

Pure PLA shows an increase of the modulus after 1 year of soil burial (see Table 1 & 6). This change is due to the drying treatment at 105°C before the actual burial test, which also introduces crystallization, thus enhancing the materials stiffness.

Table 6: Flexural properties of wod-PLA-composites after TMC exposure for 32 weeks.

	Stiffness		Strength		Strain at max. S.	
	[GPa]	stdev	[MPa]	Stdev	[%]	stdev
Pure PLA, Simlång soil	4.0	0.2	110	3.0	3.0	0.10
Furfurylated wood, Simlång soil	3.3	0.1	12.3	0.4	0.40	0.02
Furfurylated wood, Compost	2.5	0.4	10.1	1.1	0.43	0.07
Furfurylated wood, Forest	3.4	0.1	12.6	0.4	0.39	0.02
Unmodified wood PLA composites	Samples had too poor strength to be handled for testing					

Table 7: Charpy impact strength of wood-PLA-composites.

	Charpy impact [kJ/m^2]	Stdev
Pure PLA, Simlång soil	18.7	2.0
Furfurylated wood, Simlång soil	0.8	0.2
Furfurylated wood, Compost	0.7	0.1
Furfurylated wood, Forest	0.8	0.1

3.5 Field performance of CAP-WPCs (wood-CAP-composites)

In-ground stake test (EN 252)

The performance after two years in the Borås field is shown in table 8. The pine control stakes have an index of decay (IoD)of 83, meaning that they are either severely decayed or have failed. The low level CCA reference stakes are slightly decayed (IoD of 23) and the high CCA level reference stakes are all sound. Stakes from an extra set of WPCs with higher wood content (70% instead of 60% as for the rest, which meant partly non-polymer-imbedded wood particles) have all failed, whereas the control WPC stakes (with 60% unmodified wood content) are sound or slightly decayed. Except for one WPC stake from furfurylated wood which was showed signs of slight decay, all WPC stakes from modified wood were sound.

Table 8: Index of Decay after two years in the Borås field.

Test material	**Index of Decay (0-100%)**	
	1 year	**2 year**
<u>Pine sapwood specimens</u>		
Pine sapwood control	55	83
CCA Reference, 2 kg/m^3	8	23
CCA Reference, 9 kg/m^3	0	0
<u>Extruded WPCs with CAP as matrix</u>		
70% Unmodified pine	53	100
60% Unmodified pine	17	19
60% Furfurylated radiata	0	3
60% ThermoWood D spruce	0	0
60% Acetylated pine	0	0

Marine field test (Resistance to marine borers according to EN 275)

All test specimens were completely overgrown with fouling organisms and the dominating fouling species were *Ciona intestinalis* (sea squirt), *Mytilus edulis* (blue mussel), *Polysiphonia* sp. (brown slime algae) and *Pomatoseros triqueter* (keel worm).

The resistance to marine borers during the first year of exposure is shown in table 9. All pine sapwood controls were destroyed and all control WPC samples were severely attacked by shipworm (*Teredo navalis*) attack. However, all WPCs based on CAP and modified wood, both acetylated and heat treated, are completely sound.

Table 9: Condition of samples (25 x 75 x 200 mm) after one year of exposure on test rigs in the bay outside Kristineberg Marine Research Station.

	Rating of attack by		Overall
	Crustaceans *(Limnoria l.)*	**Teredinids (T. navalis)**	**Rating**
Pine sapwood specimens			
Pine sapwood control	0.4	4.0	Failed
Extruded WPCs with CAP as matrix			
60% Unmodified pine	0.0	3.0	Severe
60% ThermoWood D spruce	0.0	0.0	Severe
60% Acetylated pine	0.0	0.0	Severe

4. CONCLUSIONS

- The mechanical performance of WPCs from modified wood and bioderived thermoplastic matrix (CAP and PLA) and are equal to or better than WPCs from unmodified wood and CAP (or PLA).
- The moisture sorption is slow and the resulting EMCs are much lower for modified than for unmodified wood-CAP-Composites.
- The WPCs from CAP and modified wood, especially from acetylated and heat treated, are highly decay resistant in laboratory decay tests (soil-bock tests and TMC tests).
- Early field test results seem to support the results from laboratory decay tests.

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