EFFECTS OF WATER SOAKING–DRYING CYCLES ON THERMALLY MODIFIED SPRUCE WOOD–PLASTIC COMPOSITES

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Abstract. The overall aim of this work was to gain more insight on the potential of modified wood (TMW) components for use in wood–thermoplastic composites (WPCs). Laboratory-scale TMWPCs were produced, and the effects of severe water soaking–drying cycles on the samples were studied. Water sorption behavior and resulting dimensional and micromorphological changes were also studied, and the results were compared with those of unmodified wood–plastic composites (UWPCs) used as control. The TMW was prepared by cutting a spruce board into half and subjecting one-half to an atmosphere of superheated steam at atmospheric pressure with a peak temperature of 210°C, with the other unmodified wood (UW) half as a control. The TMW and UW components were then prepared by a Wiley mill and thereafter sifted into smaller (mesh 0.20-0.40 mm) and larger (mesh 0.40-0.63 mm) size fractions. A portion of the wood components were also subjected to hydrothermal extraction (HE). Composite samples with these different wood components, polypropylene (PP) matrix, and maleated PP (MAPP) as coupling agent (50/48/2 wood/PP/MAPP ratio by weight) were then prepared by using a Brabender mixer followed by hot pressing. The matching micromorphology of the composites before and after the soaking–drying cycles was analyzed using a surface preparation technique based on ultraviolet-laser ablation combined with scanning electron microscopy. The results of the water absorption tests showed, as hypothesized, a significantly reduced water absorption and resulting thickness swelling at the end of a soaking cycle for the TMWPCs compared with the controls (UWPCs). The water absorption was reduced with about 50-70% for TMWPC and 60-75% for HE-TMWPC. The thickness swelling for TMWPCs was reduced with about 40-70% compared with the controls. Similarly, the WPCs with HE-UW components absorbed about 20-45% less moisture and showed a reduced thickness swelling of about 25-40% compared with the controls. These observations also were in agreement with the micromorphology analysis of the composites before and after the moisture cycling which showed a more pronounced wood–plastic interfacial cracking (de-bonding) as well as other microstructure changes in the controls compared with those prepared with TMW and HE-UW components. Based on these observations, it is suggested that these potential bio-based building materials show increased potential durability for applications in harsh outdoor environments, in particular TMWPCs with a well-defined and comparably small size fractions of TMW components.

Keywords: thermally modified wood (TMW), wood–plastic composite (WPC), water absorption, dimensional stability, dynamic mechanical analysis (DMA), micromorphology, scanning electron microscopy (SEM), UV-laser ablation.

INTRODUCTION

Wood–plastic composites (WPCs) consist of wood components combined with a thermoplastic polymer matrix. The wood components usually originate from the residues created during the mechanical processing of solid wood, eg sawdust, wood shavings, and flour. The matrix is usually one of the so-called polyolefins such as polypropylene (PP) or high-density polyethylene. These raw materials are compounded and thermoformed using extruders, usually in proportions of approximately 50/50 by weight, together with a compatibilizer or coupling agent, at temperatures high enough to melt the plastic, but below 200°C to prevent unacceptable degradation of the wood (Clemons et al 2013). Extruded WPCs are mainly used in outdoor building applications such as decking, railings, roofing, and window profiles (Clemons et al 2013). In exterior applications, WPCs may be exposed to prolonged wetting- and drying-out conditions and variations in humidity. Water can mainly be present in WPCs in the lumen or the cell walls of the wood component, also in the pores of the matrix, or in the gaps/cracks between the matrix and the wood component. The MC in WPCs is normally very low after the

manufacturing process; however, it can increase after exposure to moist conditions as the material attempts to reach an equilibrium with its surroundings, through moisture absorption in the wood components. The WPCs will swell as the wood components absorb water, whereas will shrink during drying the wood. The repeated wetting and drying cycle will create inner stresses and subsequently the formation of cracks at the interface between the wood and matrix (Segerholm et al 2007; Westin et al 2008). Crack formation can lead to the development of free volume (voids) that can be filled with water. An excessively high MC can lead to an increased risk of biodeterioration, although for WPCs, the MC of the wood component normally needs to be higher than 25% to support fungal growth (Ibach et al 2016). This biodeterioration arises because of the hygroscopic character of wood. To improve the moisture-related properties of WPCs, including reducing water uptake, enhancing dimensional stability and reducing biodegradation, the lignocellulosic component can be modified to reduce its susceptibility to moisture. In addition, modification of the lignocellulosic component generally alters the interface between the polar wood components and nonpolar polymeric matrix (Arwinfar et al 2016).

Although there are many ways in which wood can be modified, including chemical and impregnation methods, thermal modification is the most established process used at commercial scale (Hill 2006). In thermal modification, the hygroscopicity of wood is reduced by the partial degradation and alteration of the chemical components of the wood cell wall that results in a decrease in the number of potential sorption sites (Hill 2006). The use of the by-products or residues from modified wood processing can increase the application value for thermally modified wood (TMW). The wood by-products from wood processing might otherwise go to combustion. To the best knowledge of the authors of this study, at this time, Lunacomp produces the only commercial WPCs prepared from TMW by-products that are on the market (Lunawood 2017). There have, however, been several research studies on WPCs incorporating TMW components. Ayrilmis et al (2011) investigated the influence of modified wood on the dimensional stability of WPCs and found reduced thickness swelling and water absorption for WPCs with TMW components compared with WPCs with unmodified wood (UW) components. In another study, Butylina et al (2011) observed improved tensile and flexural strength for WPCs with TMW fibers than for WPCs with wood flour or pellets. Flexural strength was also observed to be improved for WPCs with thermally modified sawdust together with polylactates (Butylina et al 2010) and PP (Kuka et al 2016), compared with composites with untreated sawdust. The water uptake was found to be reduced for composites with polylactates and thermally modified sawdust compared with composites with unmodified sawdust, after 2 wk (Segerholm et al 2012a) and 4 wk (Butylina et al 2010) in water. Also, a long-term water sorption showed similar trends for composites with PP and thermally modified beech (Hosseinihashemi et al 2016). Optimization of the WPC process, screw speed of the extruder, was also shown to be important for the properties of the final WPCs (Askanian et al 2015).

Another approach to improve the dimensional stability of WPCs is to reduce the water uptake of the wood components by liquid hot water extraction (Hosseinaei et al 2012; Pelaez-Samaniago et al 2013; Ozdemir et al 2014). This is a process that involves extracting mainly the polysaccharides (hemicelluloses) (Hosseinaei et al 2012; Ozdemir et al 2014) using hot water at a temperature of around 140°C. The removal of water-soluble wood compounds and extractives has also been shown to improve interfacial interactions between the nonpolar polymer matrix and the more-polar wood component (Hosseinaei et al 2012). The liquid hot water extraction of wood components for WPCs has been investigated in southern yellow pine (Hosseinaei et al 2012) and ponderosa pine (Pelaez-Samaniago et al 2013). However, little-or-no research has been conducted on how the combined effects of thermal modification and liquid hot water or hydrothermal extraction (HE) affect the performance of WPCs.

Therefore, this study aims to gain more insight on the potentials of TMW and hydrothermally extracted components for use in WPCs and effects of water soaking–drying cycles. This is a

new type of potential bio-based building material here defined as TMWPCs. The hypothesis is that TMWPCs would be less affected by repeated moisture exposure compared with conventional unmodified WPCs (UWPCs) because of the decreased hygroscopicity of the TMW components and also possibly because of improved interfacial properties between the TMW components and the thermoplastic matrix. In addition, an approach for modification of the wood components based on HE was investigated. More specifically, the objective of this work was to expose laboratory-scale TMWPCs and UWPCs, both nonextracted and hydrothermally extracted, to a series of water soaking–drying cycles and study the effect on their water sorption behavior and resulting dimensional and micromorphology changes.

MATERIALS AND METHODS

Materials

Raw material preparation. Wood components from both UW and TMW Norway spruce (*Picea abies* Karst.) were prepared from 1200-mm long board, obtained from a saw mill in Eastern Finland, with a dry density of about 387 kg/m^3. The board was first cut in half and one-half was thermally modified and the other half was used as the UW control. The thermal modification of the board was carried out in an oven in an atmosphere of superheated steam at atmospheric pressure. The process included first a drying phase at 103°C for 48 h before modification, the heating phase, with the temperature increasing from 105°C over a period of 3.5 h (30°C every 15 min), then a treatment phase having a peak temperature of 210°C for 3 h, and also an additional drying phase at 103°C for 12 h after the modification. The mass loss as a result of the thermal modification was about 5%. Both the UW and the TMW boards were cut (across the grain) into ∼5-mm strips with a band saw and milled with a Wiley mill no.2 (mesh size 1.0 mm) and then sifted into two different size fractions. The larger size fraction comprised components of mesh size between 0.40 and 0.63 mm (samples denoted 1) and the smaller size fraction comprised components of mesh size between 0.20 and 0.40 mm (samples denoted 2).

One sample group from each size fractions (ie samples 1 and 2) of the UW and TMW components was hydrothermally extracted in an air-bath digester (Haato Oy, 16140-538, Haato Oy, Finland) with a peak temperature of 140°C for 1 h. The air-bath digester included several rotating autoclaves (2.5 dm^3) heated with hot air and were filled with deionized water and wood components (solid to liquid ratio 1:25 g/g). After treatment, the wood components were filtered under vacuum through filter paper grade 4 (GE Whatman cat no.: 1004-240) and oven-dried (60°C, 24 h). The mass loss after HE can be seen in the Table 1 together with the sample information. A similar wood component material was also used in a parallel study, researching water vapor sorption properties (Lillqvist et al 2019).

The size distribution of the different sample groups was determined using Mastersizer 2000 (Malvern Instruments Ltd., Worcestershire, UK),

Table 1. Description of the wood components used for the composite samples showing the wood component sieve size, mass loss from the thermal modification, and mass loss from the HE (after thermal modification for TMW samples).

Name	Wood component (mesh sieve size) (mm)	Thermal modification	Mass loss thermal modification (%)	HE	Mass loss HE (%)
UW1	0.40-0.63	—	—	—	—
HE-UW1	0.40-0.63	—	—	X	7.7
UW2	0.20-0.40	—	—	—	—
HE-UW2	0.20-0.40	—	—	X	8.2
TMW1	0.40-0.63	X	4.7	—	—
HE-TMW1	0.40-0.63	X	4.7	X	7.4
TMW2	0.20-0.40	X	4.7	—	—
HE-TMW2	0.20-0.40	X	4.7	X	8.0

HE, hydrothermal extraction; TMW, thermally modified wood; UW, unmodified wood.

pump speed 2000 rpm. For each analysis, about 200-300 mg of dried components was used and dispersed in 900 mL distilled water. A refractive index of 1.53 (for sawdust) and density of 0.5 cm^3/g were used as input data. The component size was determined through the measurement of the volume of the specific components, and then the size was obtained from the diameter of a sphere with the same volume.

Composite preparation. The matrix consisted of PP (HE125MO Borealis, Vienna, Austria), with a density of 908 kg/m^3 and melt flow rate of 12 g/10 min. MAPP (G-3015 Eastman™, Eastman Chemical Company) was used as compatibilizer. The final composition of the mix was 50% (12.5 g) wood, 48% (12 g) PP, and 2% (0.5 g) MAPP by weight.

The composites were mixed in a Brabender Viscometer Plasti-corder PLE 651 (Brabender GmbH & Co, Duisburg, Germany) having roller-blade type impellers at 190°C, using a rotation speed of 30 rpm for 5-7 min while adding the different constituents, and a speed of 70 rpm for about 2 min during final mixing. After the mixing, smaller lumps were scraped off the mixing device and 7.6-7.7 g of compounded material were placed in a mold of dimensions 60 mm × 60 mm × 2 mm) to be pressed, giving a density of about 1056-1070 kg/m^3. The hot pressing was carried out using a force of 150 kN at 190°C for 10 min, followed by a conditioning step at a temperature of about 20°C for 2 min, applying the same pressure. Smaller samples were prepared from the composite plates for water sorption, swelling, micromorphology, and dynamic mechanical analysis, using a table top band saw.

Water Absorption and Thickness Swelling in Three Soaking–Drying Cycles

Composite samples with dimensions 15 mm × 5 mm × 2 mm (replicated five times) were exposed to three soaking–drying cycles, 39 d in total. The soaking was done in distilled water for 12 d. After each 12 d of soaking, the samples were dried in a conventional ventilated oven at 50°C for 24 h. The water was changed every 2-3 d within the cycle. The MC was calculated based on the initial sample mass and the mass of the water-soaked sample. The samples were not dried after the hot press, and the initial MC of the WPCs was between 0.3% and 0.5% before the soaking–drying cycles. Also, the dimensional change in terms of thickness was measured during the soaking and drying procedures.

Micromorphology Analysis

Samples of about 15 mm × 3 mm × 2 mm were cut using a UV excimer laser ablation (248-nm wavelength) technique to prepare suitable surfaces for the micromorphology analysis. The ablation was obtained using a krypton fluoride exciplex laser (PM-886, GSI Lumonics, Northwille, MI) (Wålinder et al 2009). The samples were further sputter-coated (Cressington Sputter coater 108 auto) with gold for 30 s before analysis with a scanning electron microscope (TM-1000, Hitachi High-Technologies Corporation, Japan). The samples were exposed to the same three cycles of soaking–drying as described previously. Images were taken in dry state at three different magnifications of (300×, 500×, and 1000×, respectively) before and after the soaking–drying cycles of samples 1 and 2. The images were observed, compared, and evaluated by the bare eye.

RESULTS AND DISCUSSION

Wood Component Size Distribution

The size fraction distribution of the sieved components is as shown in Fig 1. The larger size fractions 1 resulted in smaller sized components after the HE, especially for the TMW components. This was not the case for the smaller size fraction 2.

Water Absorption and Thickness Swelling

The results from the three soaking–drying cycles are presented in Fig 2. UWPCs (controls) showed the highest MC of all samples in the study, the

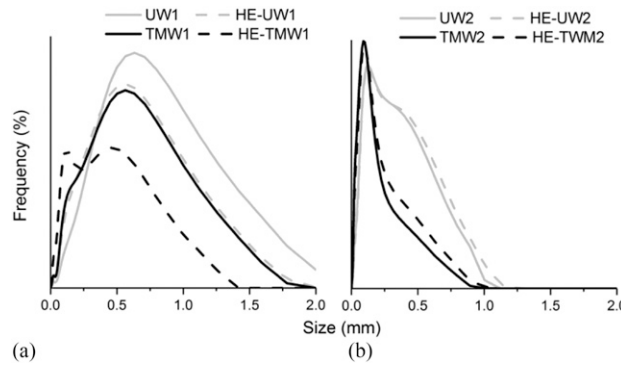

Figure 1. The size fraction distribution of the different wood components used for the composite samples; unmodified wood (UW), thermally modified wood (TMW), and both nonextracted and hydrothermally extracted (HE), where (a) shows the larger size fraction 1 (from mesh 0.40 to 0.63 mm) and (b) the smaller size fraction 2 (from mesh 0.20 to 0.40 mm).

larger fraction UW (ie sample 1) components showed higher MC values than those of the smaller UW (ie sample 2). These results imply that larger wood components would lead to higher swelling–drying stresses and cause cavities/cracks in the composite leading to higher moisture uptake. Larger wood component size fraction can also lead to an increase in bond formation due to larger contact area between the wood component and the polymer matrix of the composite. A reduction in the MC was seen for the HE-UWPCs, which was more pronounced during the first day of cycle 1. The HE-UWPCs absorbed about 20-45% less moisture than the controls at the end of the soaking cycles. The reduced MC is in agreement with other studies on water absorption for WPCs consisting of various HE pines and PP (Hosseinaei et al 2012; Pelaez-Samaniago et al 2013; Ozdemir et al 2014). The TMWPCs showed a much lower MC than the controls, a reduction with 50-70%. This was expected because of the reduced hygroscopicity of the wood resulting from the thermal modification and has also been observed in other studies of WPCs with thermally modified spruce (Segerholm et al 2007), eucalyptus (Ayrilmis et al 2011), and alder (Tufan et al 2016). The level of the water absorption or MC reduction will be dependent on eg the wood species and thermal modification process (Esteves and Pereira 2009). The HE-TMWPCs absorbed about 60-75% less moisture than the controls. The HE-TMWPCs showed only minor

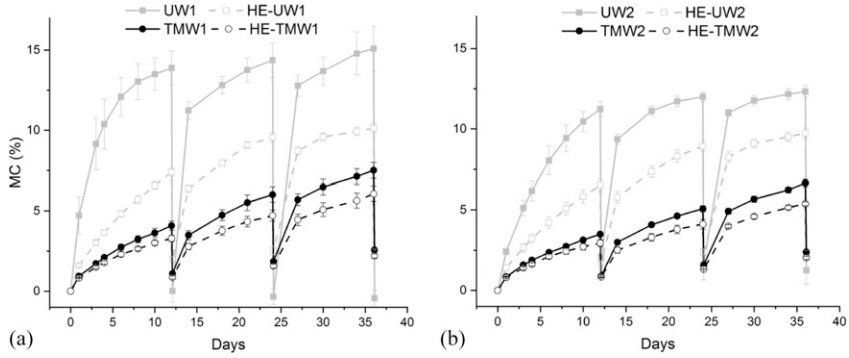

Figure 2. Water uptake expressed as MC in the composite samples vs the three soaking–drying cycles. Each value is based on an average of five replicates, including SD, for samples with unmodified wood (UW), thermally modified wood (TMW), nonextracted, and hydrothermally extracted (HE) components where (a) shows the composite samples with the larger size fraction (denoted 1) and (b) shows the smaller size fraction (denoted 2).

moisture absorption changes as a result of water soaking–drying, although the difference seemed to increase for soaking–drying cycles 2 and 3. This gradual moisture absorption increase could be the result of permanent structural changes happening during the first soaking–drying. Such information is important for the long-term performance and should be further investigated.

The rate of sorption was also higher for the controls in comparison with HE-UWPCs, and the difference was more pronounced for soaking–drying cycle 1, as presented in Fig 2. The resoaking in cycle 2 and 3 was, compared with cycle 1, faster for the HE-UWPCs and also for the TMWPCs. The rate of sorption of the TMWPCs was even lower and increased almost linearly from cycle 1 to cycle 3. The rate of sorption was generally lower for the composites with smaller size fraction components. This is because smaller size components are better dispersed in the matrix, implying less connectivity between each other, which probably led to slower wicking and thereby slower water uptake (Clemons et al 2013). It is obvious from the results in Fig 2 that the samples had some MC after the drying steps, with the exception of those with the larger fraction UW components which showed a faster drying process as the MC was around 0% after the drying. The soaking–drying conditions could be related to real scenarios where a WPC material is used in eg a window profile exposed to water soaking for several days, followed by dry weather with sunshine. In such a situation, the TMWPCs would absorb less water and be a better solution than UWPCs. In addition, the MC was still increasing during the end of cycle 3, for all samples, which means that the EMC was not yet reached. For a better understanding and prediction of the long-term effects on WPCs from water soaking–drying, future work should include more soaking–drying cycles.

The dimensional stability of the composites was measured in terms of thickness swelling. Figure 3 displays the thickness swelling of the samples during the water soaking experiments and also shows the irreversible swelling observed after each drying step. The dimensional stability was much improved for the TMWPCs compared with the controls. The HE-UWPCs also showed much lower swelling than the controls, a reduction with 25-40% at the end of the soaking cycles, where composites with larger fraction UW components showed the highest swelling. After the three cycles, the maximum swelling value of about 8.5% (0.17 mm) was observed for the samples with the larger fraction UW components, whereas the lowest value of about 3% was observed for those with the larger fraction TMW components. The swelling for various TMWPCs was reduced with about 40-70% at the end of the cycles compared with the controls. There were smaller variations in thickness swelling among the TMWPCs and interestingly, the composites with HE-TMW components showed a somewhat

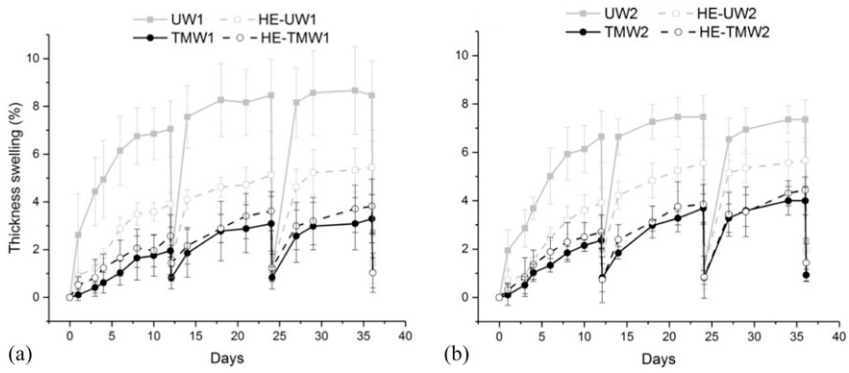

Figure 3. Thickness swelling of the composite samples for the three soaking–drying cycles. Each value is based on an average of five replicates, including SD. For sample denotations, see Fig 2.

higher swelling in the wet state than those with TMW components. The smaller size fraction showed lower swelling values in the wet state for UWPCs, whereas the HE-UWPCs showed similar values. Conversely, the TMWPCs showed higher swelling values for smaller size fraction than the ones with larger size fraction. Also, the irreversible swelling in the dry state after soaking was smaller for the samples with smaller fraction TMW and smaller fraction HE-TMW samples meaning better stability. The thickness was much reduced as a result of drying after each soaking, compared with the wet state as can be seen in Fig 3. However, an irreversible thickness increase was observed with about 2% and 1% for the controls and the TMWPCs, respectively. In addition, at the beginning of cycles 2 and 3 (after the drying step), all the samples showed consistent swelling characteristics. The reduction in swelling of TMWPCs is favorable because it will lead to fewer cracks in the material when exposed to changes in moisture conditions.

Morphology after Water Soaking

Figure 4 shows micrographs of the composite samples with smaller fractions UW, HE-UW, TMW and HE-TMW components (micrographs for the composite samples with larger fractions UW, HE-UW, TMW, and HE-TMW can be found in the Appendix). The micromorphology of the samples with UW (both larger and smaller fractions) components ie the controls were most affected by the soaking–drying cycle, shown by several pronounced wood matrix interfacial cracks, distortions of the cell walls, and cracks both in the cell wall and matrix. The cracks were observed after soaking–drying cycle 1 and slightly increased after cycle 2. This behavior was reduced for samples with HE components, where the cell walls were less damaged compared with the controls. In addition, wood matrix interfacial cracks were less pronounced in samples with HE components compared with samples containing nonextracted wood components.

Clearly, TMWPCs showed less cell wall damage and also fewer interfacial cracks compared with the WPC control sample. This is believed to be related to the reduced hygroscopicity of TMW, leading to a lower moisture uptake for the samples with TMW components. This, in turn, leads to improved dimensional stability and less swelling and shrinking of the wood components during the soaking–drying cycles. The more nonpolar character of the TMW components is further expected to improve the interfacial interaction between the matrix and the TMW components (Butylina et al 2010; Segerholm et al 2012b). Cracks that appeared at the interface between the polymer and the wood components were observed after cycle 1 but were more clearly visible after cycle 2. These results are in line with a previous study which showed that delamination and cracking occurred after the artificial aging (14 d water soaking followed by 18-h drying) of WPCs with unmodified Scots pine but was much improved for WPCs with thermally modified Norway spruce (Segerholm et al 2007).

The hot press processing condition resulted in randomly distributed wood components within the matrix, without any particular orientation, in contrast to the WPCs produced by extrusion. Wood components with their fiber direction oriented both parallel and perpendicular to the UV-laser–prepared cross section were observed. The images shown in Fig 4 compared samples with wood components perpendicular to the cross section of the image. Randomly distributed wood components within the matrix imply a more heterogeneous WPC material, compared with wood components aligned in a certain direction. This can further have influence on the swelling and mechanical properties. Also, as the wood components were separated from the extraction water through a filter paper under vacuum, they were somewhat compressed together which probably caused clumping of the components reducing the degree of dispersion in the composite. This can be overcome by adjusting the processing parameters and methodology eg by using extrusion for producing the composites, which is a more common method in the industry. However, that would require a much larger production scale.

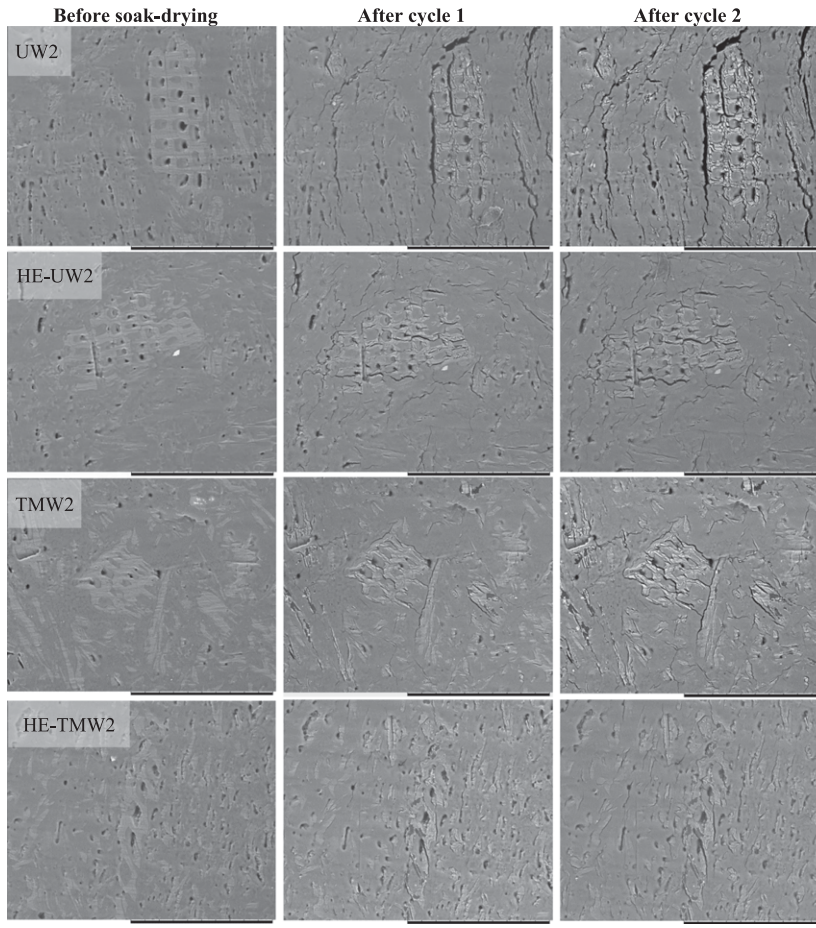

Figure 4. Matching micrographs of the composite samples with UW2, HE-UW2, TMW2, and HE-TMW2 components before and after the soaking–drying cycling. The surfaces were prepared by UV-laser ablation before the soaking–drying cycling. The scale bars correspond to 200 μm. For sample denotations, see Fig 2. HE, hydrothermal extraction; TMW, thermally modified wood; UW, unmodified wood.

CONCLUSIONS

The water absorption was reduced with 50-70% in WPCs containing TMW components, a new type of bio-based building material (here denoted as TMWPCs) compared with the WPC controls with UW. WPCs with hydrothermally extracted (HE) TMW components reduced the water absorption slightly even more, 60-75% compared with the WPC controls. In addition, the composites with HE-UWPCs showed reduced water absorption, about 20-45% compared with the controls. The rate of water absorption was much reduced for the TMWPCs samples, and also for the samples with HE components. Similarly, the thickness swelling was reduced for the TMWPC samples by about 40-70% compared with the controls. However, the HE-TMWPCs did not seem to follow any definite pattern. The HE-UWPCs showed a reduction in thickness swelling by about 25-40%. Observations of the microscopic images before and after the water soaking–drying cycles clearly showed reduced damage in the wood and wood polymer interfaces for both the samples with TMW and HE components. As an overall conclusion, it is suggested that a potential bio-based building material for the future with increased durability for applications in harsh outdoor environments may be tailored as a TMWPC with a well-defined and comparably small size fraction of TMW components. Additional

HE or other treatment of TMW components before wood polymer composite preparation should be further investigated.

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REFERENCES

Arwinfar F, Hosseinihashemi SK, Latibari AJ, Lashgari A, Ayrilmis N (2016) Mechanical properties and morphology of wood plastic composites produced with thermally modified beech wood. BioResources 11(1):1494-1504.

Askanian H, Verney V, Commereuc S, Guyonnet R, Massardier V (2015) Wood polypropylene composites prepared by thermally modified fibers at two extrusion speeds: Mechanical and viscoelastic properties. Holzforschung 69(3):313-319.

Ayrilmis N, Jarusombuti S, Fueangvivat V, Bauchongkol P (2011) Effect of thermal-treatment of wood fibres on properties of flat-pressed wood plastic composites. Polym Degrad Stabil 96:818-822.

Butylina S, Martikka O, Kärki T (2010) Comparison of water absorption and mechanical properties of wood–plastic composites made from polypropylene and polylactic acid. Wood Mater Sci Eng 5(3-4):220-228.

Butylina S, Martikka O, Kärki T (2011) Properties of wood fibre-polypropylene composites: Effect of wood fibre source. Appl Compos Mater 18(2):101-111.

Clemons C, Rowell RM, Plackett D, Segerholm BK (2013) Chapter 13. Wood/non wood thermoplastic composites. Pages 473-508 *in* RM Rowell, ed. Handbook of wood chemistry and wood composites, 2nd edition. CRC Press, Boca Raton, FL.

Esteves BM, Pereira HM (2009) Wood modification by heat treatment: A review. BioResources 4(1):370-404.

Hill CAS (2006) Wood modification: Chemical, thermal and other processes. John Wiley and Sons, Chichester, England.

Hosseinaei O, Wang S, Taylor AM, Kim J-W (2012) Effect of hemicellulose extraction on water absorption and mold susceptibility of wood-plastic composites. Int Biodeterior Biodegrad 71:29-35.

Hosseinihashemi SK, Arwinfar F, Najafi A, Nemli G, Ayrilmis N (2016) Long-term water absorption behavior of thermoplastic composites produced with thermally treated wood. Measurement 86:202-208.

Ibach R, Sun G, Gnatowski M, Glaeser J, Leung M, Haight J (2016) Exterior decay of wood–plastic composite boards: Characterization and magnetic resonance imaging. Forest Prod J 66(1):4-17.

Kuka E, Cirule D, Kajaks J, Andersone I, Andersons B (2016) Wood plastic composites made with thermally modified birch residues. Int Wood Prod J 7(4):225-230.

Lillqvist K, Källbom S, Altgen M, Belt T, Rautkari L (2019) Water vapour sorption properties of thermally modified and pressurised hot-water-extracted wood powder. Holzforschung 73(12):1059-1068.

Lunawood (2017) www.lunawood.com/composite (26 February 2018).

Ozdemir F, Ayrilmis N, Kaymakci A, Kwon JH (2014) Improving dimensional stability of injection molded wood plastic composites using cold and hot-water extraction methods. Maderas Cienc Tecnol 16(3):365-372.

Pelaez-Samaniago MR, Yadama V, Lowell E, Amidon TE, Chaffee TL (2013) Hot water extracted wood fiber for production of wood plastic composites (WPCs). Holzforschung 67(2):193-200.

Segerholm BK, Ibach RE, Westin M (2012a) Moisture sorption, biological durability, and mechanical performance of WPC containing modified wood and polylactates. BioResources 7(4):4575-4585.

Segerholm BK, Rowell RM, Larsson-Brelid P, Wålinder MEP, Westin M, Alfredsen G (2007) Improved durability and moisture sorption characteristics of extruded wood-plastic composites made from chemically modified wood. Pages 253-258 *in* 9th International Conference on Wood and Biofiber Plastic Composites, May 21-23, 2007, Monona Terrace Community and Convention Center, Madison, WI.

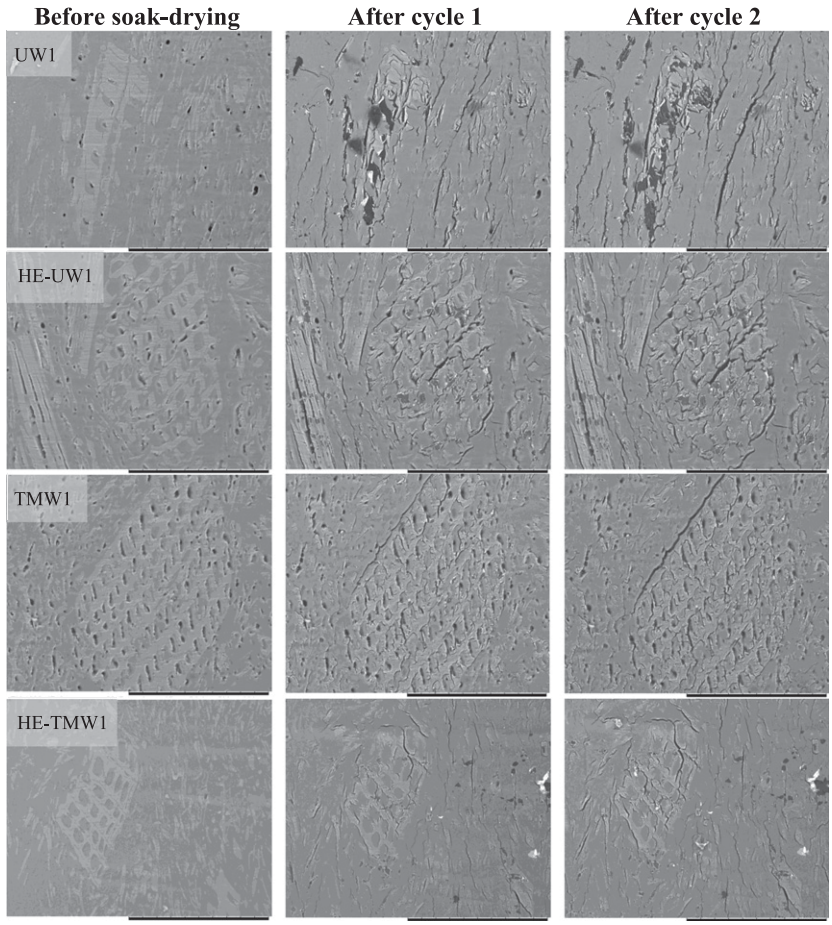
Segerholm BK, Vellekoop S, Wålinder ME (2012b) Process-related mechanical degradation of the wood component in high-wood-content wood-plastic composites. Wood Fiber Sci 44(2):145-154.

Tufan M, Güleç T, Peşman E, Ayrilmis N (2016) Technological and thermal properties of thermoplastic composites filled with heat-treated alder wood. BioResources 11(2):3153-3164.

Wålinder M, Omidvar A, Seltman J, Segerholm K (2009) Micromorphological studies of modified wood using a surface preparation technique based on ultraviolet laser ablation. Wood Mater Sci Eng 1-2:46-51.

Westin M, Larsson-Brelid P, Segerholm BK, van den Oever M (2008) Wood plastic composites from modified wood. Part 3. Durability of WPCs with bioderived matrix. *In* Proc. 39th Annual Meeting of the International Research Group on Wood Preservation (IRG), May 25-29, 2008, IRG/WP 08-40423, Istanbul, Turkey. 17 pp.

APPENDIX



Appendix Figure. Matching micrographs of the composite samples with UW1, HE-UW1, TMW1 and HE-TMW1 components before and after the soaking-drying cycling. The surfaces were prepared by UV-laser ablation before the soaking-drying cycling. The scale bars correspond to 200 μm. HE, hydrothermal extraction; TMW, thermally modified wood; UW, unmodified wood.