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Effect of hot water extracted hardwood and softwood chips on particleboard properties

Abstract: The affinity of particleboard (PB) to water is one of the main limitations for using PB in moisture-rich environments. PB dimensional stability and durability can be improved by reducing the available hydroxyl groups in wood through hemicellulose removal, for example, by hot water extraction (HWE), which increases wood resistance to moisture uptake. The resulting liquid fraction from HWE is rich in hemicelluloses and can be used for chemicals and fuels, and the solid fraction is less hydrophilic. The objective of this study was to investigate the effects of HWE of softwood chips (conducted at 160°C and 90 min) and hardwood chips (160°C and 120 min) on the properties of PB panels. HWE increased compressibility and reduced springback by 34% and 44% for pine and maple chips, respectively, which positively impacted the PB properties. Water absorption of pine PB panels was lowered by 35% and that of maple PB panels by 30%, while reduction of thickness swelling was lowered by 39% for pine PB and 56% for maple PB after 24 h of immersion in water. The mechanical properties were not significantly affected.

Keywords: hemicelluloses, hot water extraction, lignin, particleboard, springback, wood-water affinity

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Introduction

Particleboard (PB) is a nonstructural wood composite used widely in the building trades. However, the affinity of PB to water is a limitation for utilization under moisture-rich conditions, which also affects its dimensional stability and durability. Hemicelluloses in the wood cell wall are the most hydrophilic wood constituents (Skaar 1972). Thus, one of the strategies for quality improvements of wood composites is the extraction of hemicelluloses through thermochemical processes (Pelaez-Samaniego et al. 2013). To this purpose, hot water extraction (HWE) is preferred, as it does not need additional chemicals (Garrote et al. 1999).

Hemicellulose removal may also affect wood properties, such as surface area and pore size (Kobayashi et al. 2009; Duarte et al. 2011), surface roughness (Anglès et al. 1999), as well as strength, toughness, and the abrasion resistance (Kamdern et al. 2002; Shi et al. 2007). Sweet and Winandy (1999) found a negative effect of hemicellulose reduction on the wood mechanical properties after exposing wood to hot air, while the degree of polymerization (DP) of cellulose did not change (Sweet and Winandy 1999). Horn (1979) found that the strength and stiffness of press-dried pulp sheets are correlated with the amount of hemicelluloses. Klüppel and Mai (2012) showed that hemicelluloses promote wood strength under dry conditions, while lignin is responsible for maintaining wood strength under wet conditions.

HWE is an autocatalytic thermochemical process for fractionation of easily accessible sugars in lignocellulosic biomass (Amidon et al. 2008), resulting in the modification of wood properties (Boonstra and Tjeerdsma 2006; Boonstra and Blomberg 2007). During HWE, hemicelluloses are depolymerized by hydrolysis into monomers and oligomers (Garrote et al. 1999) and removed as aqueous solution. The degree of crystallinity of cellulose is increased (Bhuiyan et al. 2000) and both cellulose and lignin are partially depolymerized (Garrote et al. 1999). Lignin may also be subjected to plasticization (Bouajila et al. 2005), partial solubilization (Tunc and van Heiningen 2008), and condensation (Boonstra et al. 2006). These reactions alter substantially the properties and the chemical composition of

the remaining solid fraction. However, HWE does not disintegrate the physical structure of the wood particles such as a chip or a strand. HWE is an effective pretreatment prior to the production of chemicals and fuels (Amidon et al. 2008; Alvira et al. 2010) and the pretreated chips or strands have improved properties for manufacturing wood composites in terms of lower wood-water affinity followed by better dimensional stability (Boonstra et al. 2006; Mohebbi et al. 2008; Paredes et al. 2008; Sattler et al. 2008; Hosseinaei et al. 2011a; Pelaez-Samaniego et al. 2012, 2013).

A higher transverse compression after HWE could be beneficial for manufacturing wood composites by hot-pressing technology, in the course of which higher compaction ratios can be achieved at lower pressures. As a result, the mat has a higher densification and more contact between the particles promoting bond formation. Higher compaction ratios generally lead to significantly better strength and stiffness of the composites and all these effects also contribute to decreasing the springback of wood particles. Thickness recovery of densified particles is one of the reasons for breaking of the wood-adhesive bond network (Sekino et al. 1999). Thus, hemicellulose removal upon HWE is the main reason for lower springback of the panels after hot-pressing (Hsu et al. 1988). Boonstra and Blomberg (2007) also demonstrated the positive impact of thermochemical wood modification on wood densification. However, limited research has been conducted on the effects of HWE on wood compressibility and springback. The objective of this study was to understand the effect of HWE on wood properties, especially with focus on the compressibility and springback behavior of wood chips, which are targeted for PB production. The dimensional stability and mechanical properties of PB panels manufactured with HWE pretreated softwood and hardwood particles will also be a focus of the present paper.

Materials and methods

Raw materials investigated were (1) debarked Ponderosa pine (*Pinus ponderosa* Dougl. Ex Laws.) logs with small diameters from a fuel reduction treatment in central Oregon State (Pelaez-Samaniego et al. 2012) and (2) debarked sugar maple (*Acer saccharum* Marsh.) logs (obtained from Heiberg Forest Properties, State University of New York, Tully, NY). Both pine and maple logs were chipped to dimensions between 9 and 50 mm long, 14 and 20 mm wide, and 2 and 6 mm thick. The HWE of pine chips was carried out at 160°C and 90 min (Chaffee 2011) and of maple chips at 160°C and 120 min (Amidon et al. 2008; Duarte et al. 2011). The residual solid fractions after HWE were 80% for pine and 77% for maple. Unextracted pine (P_{unex}), HWE pine (P_{HWE}), unextracted maple (M_{unex}), and extracted maple (M_{HWE}) materials were comminuted in a hammer mill (Bliss

Industries) equipped with a 6.35 mm diameter holes screen. Particle size distribution was determined as per ASTM D5644-01. Approximately 12 kg of each material were dried in an electrically heated drum-drier. Modified urea formaldehyde (UF) (Casco-Resin™ HTR01B/TD3158/2.5M, Momentive Specialty Chemicals, Inc., Columbus, OH, USA) served as binder for pressing PB.

Fourier transform infrared (FTIR) (Thermo Nicolet Nexus 670 spectrometer coupled with an ATR cell) was used to study the main changes occurring on HWE material after HWE. FTIR spectra were collected in the range of 4000 to 400 cm^{-1} with 32 scans at a spectral resolution of 2 cm^{-1} . The normalization of spectra was conducted (Faix 1991). The morphology of chip surfaces before and after HWE was observed by scanning electron microscopy (SEM; FEI Quanta 200F SEM equipment).

Compressibility and springback of the raw material were tested on wood chips. The chips were manually selected aiming at parallel direction of the grains to large surfaces. These surfaces were roughly in the radial plane, that is, with compression loading in the tangential direction. Six specimens of each raw material were prepared with a belt sander machine (200-grit sanding belt). Dimensions of the resulting prismatic specimens were approximately $11 \pm 1 \times 11 \pm 1$ mm, with 2.5 ± 0.5 mm thickness (measured in the center with a micrometer, precision 0.01 mm). Special attention was paid during sanding to obtain specimens with their larger faces as parallel as possible (thickness deviation between the edges was < 0.1 mm). The specimens were then dried at 103°C for 24 h.

Weight was determined with a laboratory balance (precision 0.001 g); initial thickness (t_o) was measured after oven drying. An Instron 3345 testing machine (equipped with a force transducer model 2519-107 and a data acquisition system) was used for compression tests on the dry (prismatic) chips at room temperature (RT). The order of testing each set of specimens was randomly selected. The loading speed of the compression head was 3 mm min^{-1} until the load reached 1 N, and the speed was then reduced to 0.5 mm min^{-1} (Ellis and Steiner 2002) and maintained until the load reached 4500 N. The pressure was released immediately after the load reached 4500 N. The load limit (4500 N) set did not intend to simulate the hot-pressing conditions of PB in a press but rather to evaluate the effect of HWE on wood compressibility in the tangential direction and springback at RT.

The thicknesses of the compressed chips (t_c) were measured immediately after compressing. Approximately 3 h after compressing, the chips were soaked in water for 2 h. Soaking for 2 h guaranteed saturation. The specimens were subsequently oven dried at 103°C for 24 h and thickness (t_r) was measured once more. The compressibility (c) of wood specimens was computed as $c = (t_o - t_c) / t_o$, and the springback (s) was determined as $s = (t_r - t_c) / (t_o - t_c)$ (Inoue et al. 2008), which is in accordance with the definition of springback suggested by Kelly (1977). SEM was employed to visualize the effect of compression. To this purpose, two compressed chips of each raw material were randomly selected, and their cross-sections were prepared under moist conditions following the method of Hoadley (2000) and dried for 24 h; multiple SEM micrographs from pretreated and untreated chips of both species were prepared.

For PB production, dry particles [moisture content (MC) $< 1\%$] of P_{unex} , P_{HWE} , M_{unex} , and M_{HWE} materials were mixed with modified UF in a rotating drum-mixer and then formed to dimensions of approximately 610×812 mm². Three panels of each material (12 in total) were hot-pressed at $143 \pm 2^\circ\text{C}$ in a hydraulic press (Siempelkamp, 3800 kN capacity) to obtain homogeneous PB (one random layer). The target density of the panels was 700 kg m³. The pressing cycle consisted of

curing the mats for 180 s at 12.5 mm thickness followed by 60 s of degassing at 16.5 mm press opening.

Water absorption (WA) and thickness swelling (TS) behavior (ASTM D1037-06a, Method A: 2-Plus-22-h immersion in water) of PB panels were evaluated. Six specimens from each panel were prepared (dimensions 152.4×152.4×12.5 mm³) and conditioned at 65% relative humidity (RH) at 20°C until saturation. The specimens were weighed, and dimensions were recorded to compute densities at the beginning of the test. The specimens were then horizontally immersed in distilled water at RT. Weight and thickness were recorded for each specimen after 2 and 24 h of immersion. The thickness was measured at five points previously labeled on each specimen: four close to the corners and one in the center. After 24-h measurements, the specimens were oven dried for 24 h to determine oven-dried weight.

Nine specimens of each formulation (305×76×12.5 mm³) were evaluated for linear expansion (LE) as a function of MC (ASTM D1037-06a) in a Russells (G-64 Elite) environmental chamber. Initially, the specimens were conditioned in the chamber at 50% RH and 20°C. When the specimens were saturated (difference in weight measured between 24-h periods was <0.5%), the RH was increased to 90% keeping the initial temperature, and weights and dimensions were recorded. Equilibrium MC (EMC) of panels and raw materials at three different RH (50%, 70%, and 90%) was tested in the environmental chamber at 20°C. This test was carried out using five specimens for each formulation (dimensions 152×75×12.5 mm³). In addition, the EMC of two samples of each raw material (~20 g of wood particles each) was also determined. The tests started at an initial setting of 50% RH. When the specimens were saturated, the RH was increased to 70% until saturation, and the specimens were weighed again. The process was then repeated with 90% RH. After this final conditioning, a specimen of each formulation was selected and small prismatic pieces were prepared intending to observe the cross-section of the panels by SEM.

Static bending, tension parallel to the surface, and tension perpendicular to the surface [internal bond (IB)] properties were evaluated following ASTM D1037-06a and ANSI A208.1-1999 PB guidelines by means of a screw-driven Instron 4466 machine equipped with a 10 kN load cell. The support span for bending was 24 times the depth of the specimen (i.e., 300 mm). For tension, the displacement was recorded in a 25-mm displacement extensometer (MTS model 634.12E-24). Speeds of testing were 6 mm min⁻¹ for static bending, 4 mm min⁻¹ for tension parallel to the surface, and 1 mm min⁻¹ for IB.

The effect of HWE on the homogeneity of density across the panels' sections was evaluated through thickness density profile. The tests were conducted with five specimens (previously conditioned at 65% RH and 20°C) of each material (dimensions 50.8×50.8×12.5 mm³) in a QMS X-ray density profiler (Model QDP-01X). The difference between the maximum and the minimum values (peaks and valleys) of each curve was taken as a measure for evaluating the impact of HWE on thickness density profile.

Results and discussion

Changes in the raw materials promoted by HWE

Particle size distributions of the P_{unex} and the P_{HWE} materials were quite similar (Figure 1). Pine showed easy-to-classify

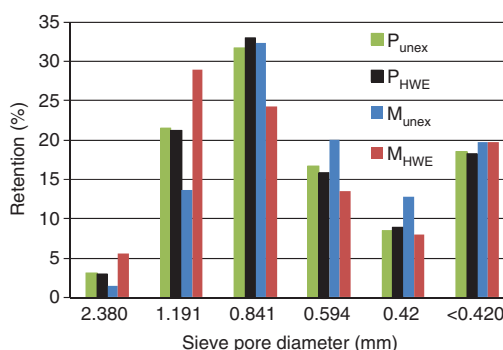


Figure 1 Particle size distribution of raw materials.

fragmented particles. M_{HWE} , on the contrary, showed a “curly” texture, making it more difficult to classify particles of this material by size.

FTIR results

Normalized FTIR spectra (Figure 2) show some important differences of the spectra of P_{HWE} versus P_{unex} and M_{HWE} versus M_{unex} . The bands at 1740 cm⁻¹ have been assigned to hemicelluloses (Kobayashi et al. 2009). The intensity of these peaks decreases after HWE, meaning that hemicelluloses were partially removed. The intensity of the broad bands at approximately 3400 to 3570 and 3230 to 3310 cm⁻¹ (see arrows) does not show important changes after HWE in both species. Since these bands are assigned to the hydrogen-bonded O-H groups in intramolecular and intermolecular cellulose, respectively (Kobayashi et al. 2009), it appears that cellulose was not degraded during HWE. The increase of the peak's intensity at 1510 and 1425 cm⁻¹ is indicative for elevated lignin contents (Kobayashi et al. 2009; Hosseinaei et al. 2011b). The peaks of both P_{HWE} and M_{HWE} become sharper at 1030 cm⁻¹, which can be attributed to condensed lignin fractions (Kobayashi et al. 2009). Discussions about the meaning of bands can be found in Owen and Thomas (1989), Faix (1991), Evans (1991), Langkilde and Svantesson (1995), or Schwanninger et al. (2004).

SEM results

SEM images of extracted and unextracted materials (Figure 3) show morphological changes on wood particle surfaces and cell walls during HWE. Both P_{HWE} and M_{HWE} display small spherical-like droplets with different dimensions (less intense in P_{HWE}). In P_{HWE} , however, these droplets tend to be agglomerated. This feature has been

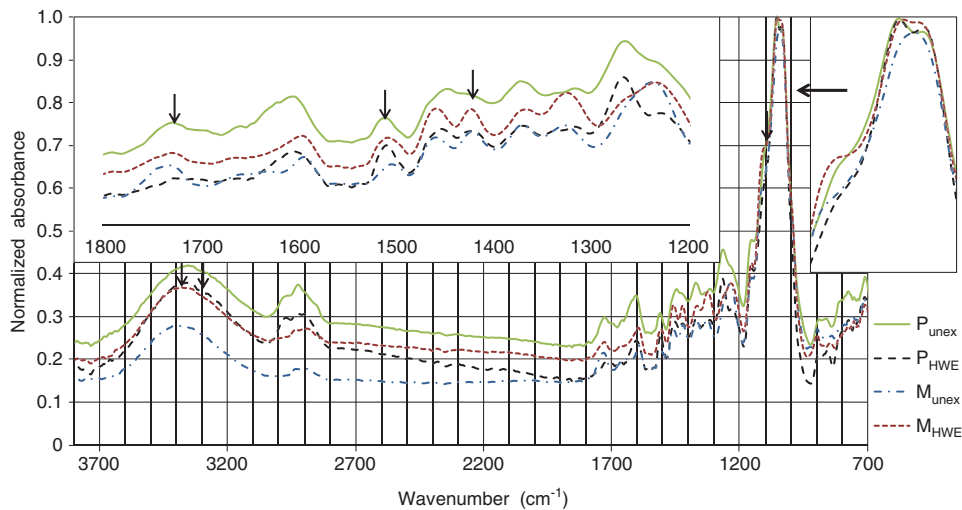


Figure 2 FTIR transmittance spectra of treated (by HWE) and untreated materials.

attributed to the coalescence and partial migration of lignin to the surface (Selig et al. 2007; Donohoe et al. 2008), which occurs also in the case of steam-explosion treatment (Marchessault 1991). Zhou (2013) referred to this type of material as lignin liquid intermediates (LLI), which are reactive at high temperatures. Therefore, it is expected that LLIs play an important role during hot-pressing of wood

composites produced with thermally modified wood. The difference in the morphology and, possibly, the amount of deposited LLI on the surfaces of pine and maple cell walls could result from the longer HWE time of maple and the properties of its lignin. Less amount of free OH_{phen} groups and greater amount of OMe groups (meaning less cross-link density) are typical for hardwood lignins, which result in

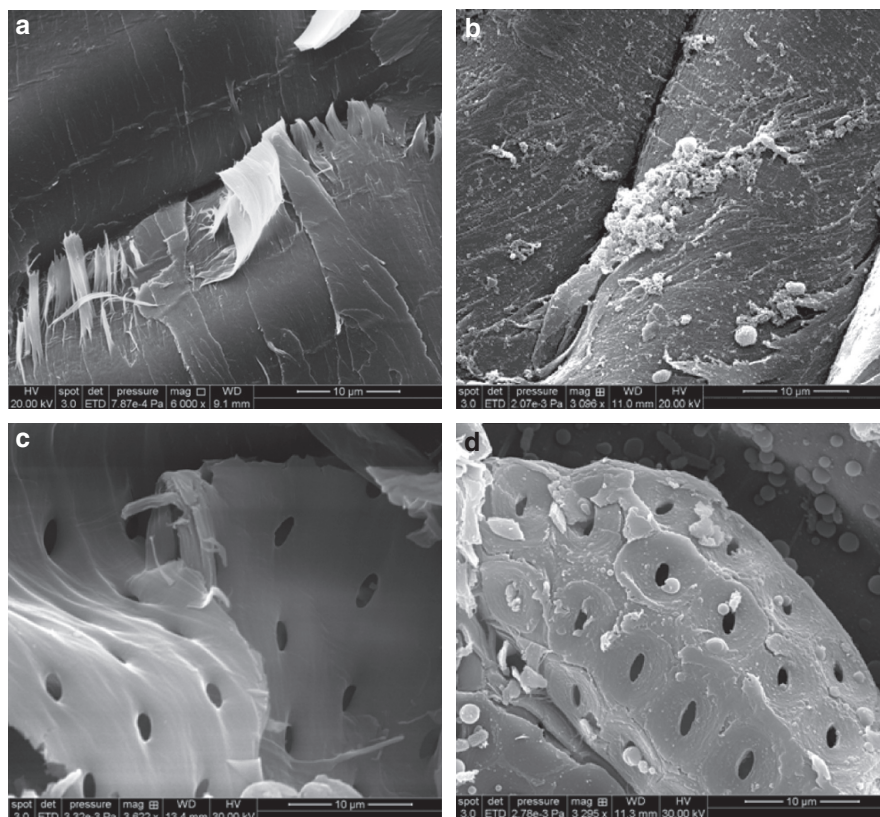


Figure 3 SEM micrographs of chips of (a) P_{unex} , (b) P_{HWE} , (c) M_{unex} , and (d) M_{HWE} . Material deposited on surfaces of treated wood (droplet-like) is believed to be lignin-rich compounds.

lower softening temperatures compared to softwood lignins (Olsson and Salmén 1992). These deposits and other derivatives of wood would deserve more study concerning the construction of composites. Lignin is known as a natural binder (Mason 1928; Byrd 1979; Winandy and Rowell 1984), bioprotecting agent (Agbor et al. 2011; Chirkova et al. 2011), and water repellent (Horn 1979; Winandy and Rowell 1984; Klüppel and Mai 2012). The relative increase of lignin in the HWE-treated material contributes to its less hydrophilicity, as observed in wood pulp (Horn 1979).

Results of compression testing of wood chips

Typical stress-strain curves of wood chips in compression are presented in Figure 4. Compressibility of HWE chips is significantly higher in the case of P_{HWE} compared to P_{unex} . Tough compressibility of M_{HWE} was approximately similar than that of M_{unex} (Table 1). On the average, Young's modulus of HWE chips is reduced by 6% (pine) and 20% (maple) (statistically significant at the $\alpha=0.05$ level). Percent strain at same stress level of 15 MPa (Figure 4) was also higher for HWE chips. This pressure level was chosen because all panels were subjected to this pressure during hot-pressing prior to reaching a maximum pressure and subsequent pressure reduction (Figure 5). On average, percent strain at the 15 MPa stress level approximately doubled for pine and increased by 16% for maple as a result of HWE (although not statistically significant at the $\alpha=0.05$ level in the case of maple chips). Accordingly, the better compressibility of HWE chips in transverse compression is demonstrated. Springback, on the contrary, was reduced by approximately 31% and 44% for HWE pine and maple, respectively. Thus, the effects of transverse deformation of wood cells on HWE chips are more permanent than in the case of natural wood.

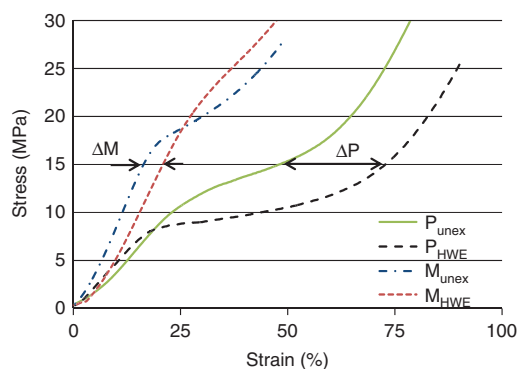


Figure 4 Typical compression stress-strain curves of prismatic chips (chips were oven dried prior compression). ΔP and ΔM refer to the relative increase of strain (as a consequence of HWE) when stress reaches 15 MPa.

Table 1 Compressibility and springback of prismatic wood chips specimens during compression tests.

Materials	Compressibility (%)	Springback (%)	Young's modulus (GPa)	Strain at 15 MPa
P_{unex}	43.3 (8.5) A	85.7 (7.2) A	2.07 (23.2) A	29.9 (18.8) A
P_{HWE}	53.1 (6.4) B	59.3 (8.2) B	1.95 (4.6) A	62.6 (4.6) B
M_{unex}	24.0 (17.7) A	89.3 (7.4) A	1.12 (10.6) A	17.1 (13.2) A
M_{HWE}	23.4 (14.0) A	49.9 (11.5) B	0.90 (16.9) B	19.9 (18.5) A

For each property, the comparison is based on means. In parentheses: coefficients of variation (%). Values with different letters indicate significant differences at the $\alpha=0.05$ level. Since the test of equality of variance was not significant, the pooled t test was used for the comparison of properties.

Compression of HWE chips induces permanent transverse deformation of wood cells, which is not recovered upon unloading, as confirmed by SEM analysis.

SEM pictures (Figure 6a-d) show that compressed chips from extracted wood remain deformed even after soaking in water and redrying. The improved springback of particles is difficult to quantify in terms of the dimensional stability of PB because of the random shape and orientation of particles in the panels and the effect of temperature and bond formation during the hot-pressing process. However, the effects are certainly positive. For comparison, the SEM micrographs of PB cross-sections are also presented in Figure 6e and f. Clearly, particles inside PB remain deformed after hot-pressing of boards, similar to the observations on prismatic chips (Figure 6a-d).

Impact of HWE on PB manufacture and properties

The pressure required for hot pressing HWE pine and maple mats is slightly less than that for mats of untreated

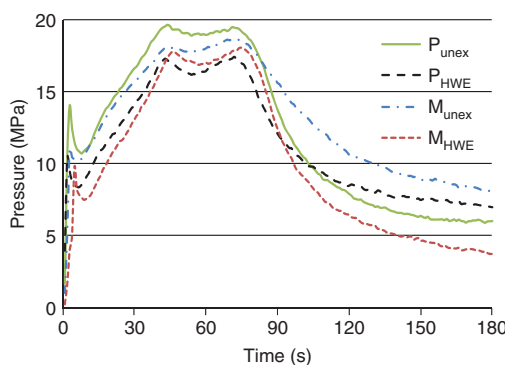


Figure 5 Typical pressure versus time curves collected during hot-pressing of PB panels.

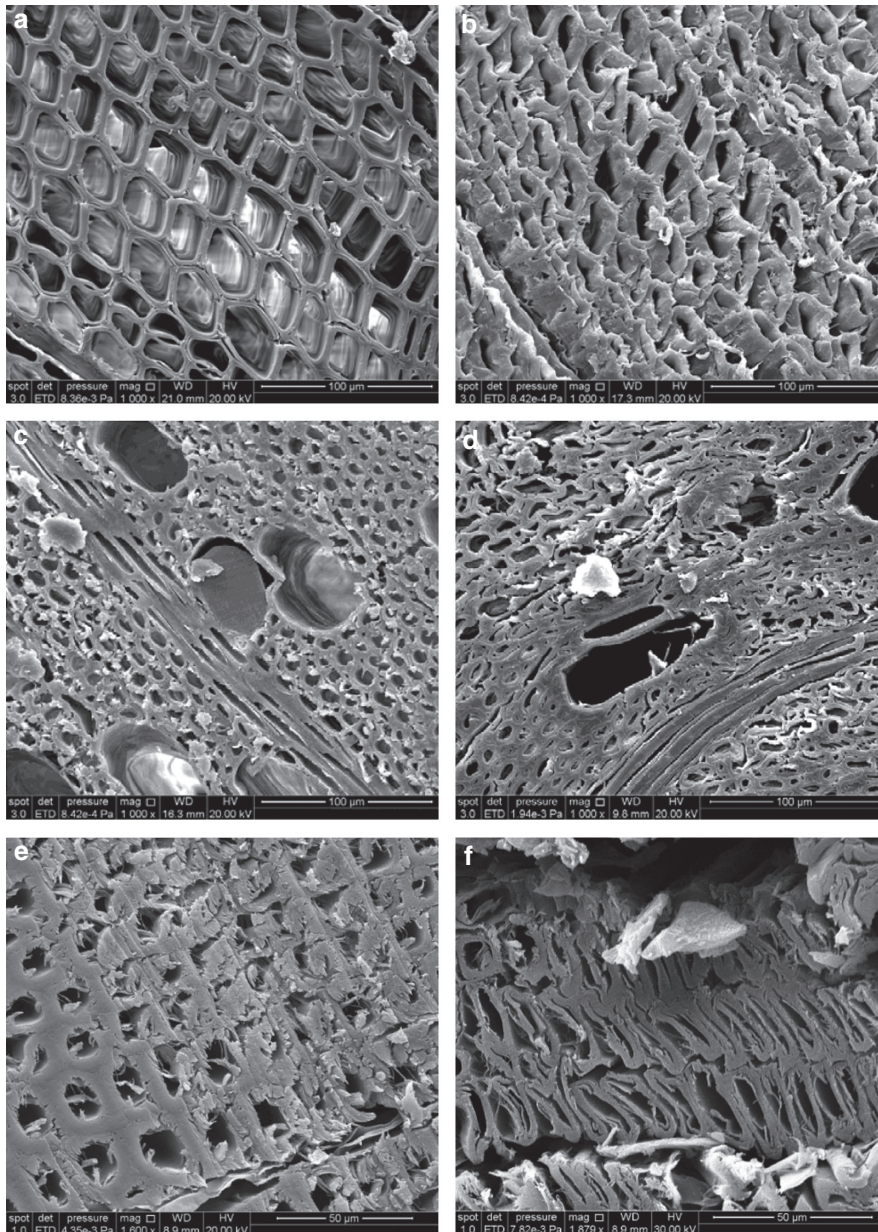


Figure 6 SEM micrographs of the cross-section of compressed prismatic chips of (a) P_{unex} , (b) P_{HWE} , (c) M_{unex} , and (d) M_{HWE} and cross-section of PB panels produced with (e) P_{unex} and (f) P_{HWE} .

materials (Figure 5). This observation is in agreement with that made based on Figure 4. This result has influence on the energy consumption during hot pressing of panels.

WA, TS, and LE as a function of MC

The surface of PB panels produced with HWE chips was smoother and dark brown in color. The resistance to water and dimensional stability were improved. Although the target density was 700 kg m^{-3} , the actual densities of PB panels with untreated materials after conditioning

(Table 2) were slightly lower than those produced with HWE materials, but this observation is statistically not significant. A lower density of PB_{unex} could result from the thickness recovery after pressing.

Specimens subjected to WA increased their weight mostly during the first 2 h of immersion in water (Table 2). WA of both P_{HWE} and M_{HWE} formulations decreased by approximately 36% and 34% after 2 h of immersion and by 35% and 30% after 24 h of immersion, respectively. This result shows that HWE elevates the barrier effect to moisture penetration into the voids leading to less moisture uptake of panels.

Table 2 WA of PB panels.

Materials	At the beginning, after conditioning		Moisture uptake (%)	
	Density (kg m ⁻³)	MC (%)	2-h immersion	24-h immersion
P _{unex}	671.1 (4.7) A	10.6 (13.5) A	148.7 (6.8) A	162.0 (6.3) A
P _{HWE}	704.2 (2.5) A	6.8 (18.5) B	95.0 (10.2) B	105.6 (9.1) B
M _{unex}	666.3 (7.4) A	15.5 (10.7) A	153.0 (5.9) A	155.4 (5.6) A
M _{HWE}	712.5 (2.9) A	10.5 (27.7) B	101.1 (8.5) B	108.2 (7.5) B

For statistical explanations, see Table 1.

TS of PB specimens measured after 24 h of water immersion (Table 3) was reduced by approximately 39% for pine and approximately 56% for maple. When the specimens were subjected to environments with RH changing from 50% to 90%, the observed TS reduction was 51% (pine) and 53% (maple). Moreover, the LE of the HWE materials was reduced by approximately 50% (Table 3). These large reductions in TS and LE are in close relation to an essential hygroscopicity decrement of HWE wood.

EMC of wood furnish and PB panels (Figure 7) illustrate that the EMCs of HWE furnishes are diminished compared to unextracted wood furnish irrespective of the RH. Similar behavior is also observed for PB panels. The EMC of PB at 90% RH is reduced by approximately 30% (pine) and 23% (maple). Comparison of EMC values between wood furnish and PB panels at similar RH levels shows that PB sorption is less intense than that of the corresponding furnish (see also Suchsland 2004).

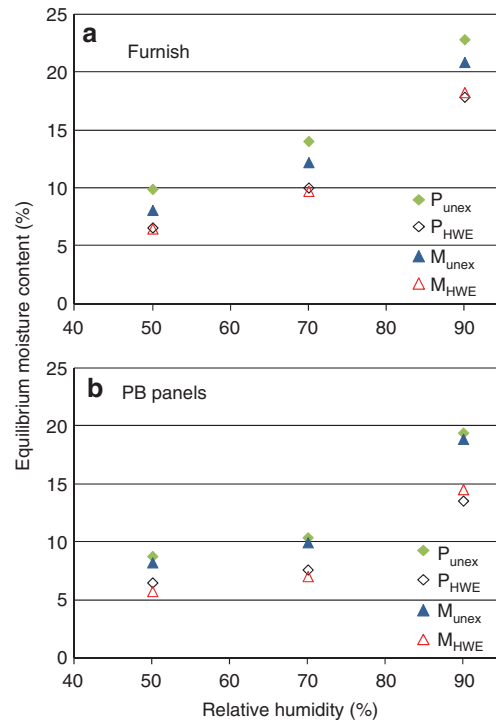
Mechanical properties of PB

The values of modulus of rupture (MOR) and modulus of elasticity (MOE) in bending are increased in formulations containing HWE wood (Table 4) and slight IB reduction is observed in pine PB. However, these changes are

Table 3 TS and LE with change in MC of PB specimens.

Materials	TS (%)		LE (%) at	
	2-h immersion	24-h immersion	RH change 50→90%	RH change 50→90%
P _{unex}	44.0 (9.8) A	48.2 (12.0) A	2.13 (6.57) A	2.12 (4.57) A
P _{HWE}	24.9 (4.7) B	29.3 (4.3) B	1.04 (8.37) B	1.03 (5.42) B
M _{unex}	62.9 (15.0) A	64.3 (15.6) A	2.06 (8.86) A	1.16 (0.49) A
M _{HWE}	23.7 (8.7) B	28.5 (8.1) B	0.97 (10.71) B	0.60 (8.58) B

For statistical explanations, see Table 1.

**Figure 7** EMC of (a) furnish and (b) PB specimens (RH was changed in an environmental chamber).**Table 4** Mechanical properties of PB specimens.

Materials	Density (kg m ⁻³)	MOR (MPa)	MOE (GPa)	IB (kPa)
P _{unex}	694.8 (2.9) B	4.7 (16.2) A	0.726 (8.5) A	461.1 (20.1) A
P _{HWE}	695.2 (7.6) B	5.5 (13.1) A	0.794 (20.5) A	419.7 (16.9) A
M _{unex}	661.8 (4.6) A	4.8 (17.1) B	0.784 (15.6) B	385.0 (9.7) A
M _{HWE}	697.6 (6.8) A	5.4 (20.4) B	1.034 (28.3) B	429.7 (16.7) B

For statistical explanations, see Table 1.

statistically not significant at the $\alpha=0.5$ level of confidence. High coefficients of variation in mechanical properties result mainly from the difficulty to guarantee homogeneous density throughout the panels. Statistically, IB of formulations containing maple is increased, showing a positive effect of HWE on this property.

Analysis of thickness density profile (Figure 8) shows that the differences between the maxima and the minima of the density profile curves were significantly reduced as a consequence of HWE. These differences were 243.8 ± 55.1 , 113.4 ± 8.3 , 103.3 ± 5.0 , and 78.2 ± 13.7 kg m⁻³ for P_{unex}, P_{HWE}, M_{unex}, and M_{HWE}, respectively. These differences are statistically significant (at the $\alpha=0.5$ level of confidence) at the species level. Therefore, HWE furnish would result in a more uniform vertical density profile than the control furnish.

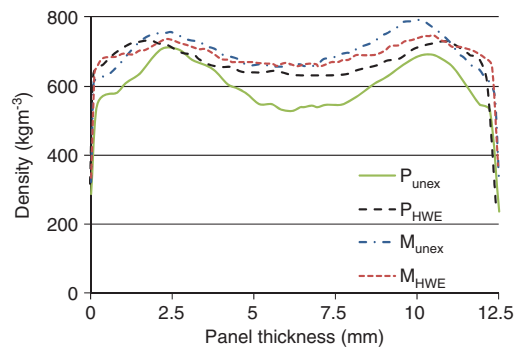


Figure 8 Typical vertical density profile curves of PB panels produced with P_{unex} , P_{HWE} , M_{unex} , and M_{HWE} .

Improvement of physical properties of PB panels through HWE without negatively affecting mechanical properties is a promising result that could impart greater durability to the composite panel and allow its use in moisture-rich environments.

Conclusions

HWE of both softwood and hardwood has substantially reduced the water affinity of wood and, consequently,

has increased the dimensional stability of PB. Composite panel mechanical properties made of HWE chips have not been affected significantly. Improvement of PB panel properties is attributed mainly to the removal of hemicelluloses, which promoted easier compression of particles, greater fiber flexibility, and permanent deformation, contributing to less springback. PBs with improved dimensional stability and moisture resistance open new markets such as for flooring and exterior trimming applications.

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