Effect of Hemicellulose Extraction on Physical and Mechanical Properties and Mold Susceptibility of Flakeboard

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Abstract

Hemicellulose is the most hydrophilic polymer of wood, and as a polysaccharide, it has potential applications in conversion to biofuels. The objective of this study was to enhance properties of flakeboard by extracting hemicellulose. Hot-water pretreatment was performed to extract hemicellulose under different temperatures (140°C, 155°C, and 170°C) and times (30 and 60 min). The flakes were blended with 5 percent liquid phenol-formaldehyde resin and 1 percent wax emulsion. The mat was pressed at 200°C for 5 minutes. The physical and mechanical properties and the susceptibility of flakeboard to mold were studied. Panels made from the hemicellulose-extracted flakes showed remarkable decreases in water absorption and thickness swelling without a decrease in mechanical properties. Resistance of the panels to the mold growth also increased with increasing mass loss due to extraction. The most severe condition of extraction (170°C, 60 min), in addition to having the lowest water absorption and thickness swelling, showed the highest mold resistance.

Fossil fuels are not renewable and cannot be considered as a long-term principal source of energy for the future. The production of bioenergy, mainly in the form of bioethanol, is a common current usage of biomass as a renewable energy source. Crops like sugar cane and corn, which are primarily used for food, are now being used for the production of bioethanol (Goldemberg 2007). Biofuel production has increased demand for crops and grains like corn and wheat, and it is also one of the reasons for the recent global rise of food prices (Buntrock 2007). To be economically feasible, the production cost of bioethanol must be lowered to make it commercially comparable with fossil fuel. In view of these issues, efforts are being made to use lignocellulosic materials such as wood and agricultural and forest residues, which are potentially cheap, to produce biofuel (Sun and Cheng 2002). Specifically, current technologies for bioenergy are using cellulose and/or hemicellulose of biomass for producing bioethanol. An alternative approach is to extract some sugars (mainly hemicellulose) from woody materials for use in the biofuel industry and to use the remaining materials (lignin and cellulose) in other applications like paper and composites.

The principal carbohydrates in wood are cellulose and hemicellulose. Hemicellulose is the second most common polysaccharide in woody plants (20% to 35% of lignocellulosic biomass). The random and amorphous structure of hemicellulose makes it easy to hydrolyze (Hu et al. 2008). Recently, the bioconversion of hemicellulose has received attention because of its possible applications in conversion to fuels and chemicals (Saha 2003).

One potential case for integrating biofuel and wood composite industry is oriented strandboard (OSB). Over 17 billion square feet of OSB were consumed in the USA in 2008, mainly in residential buildings (Essential Information for the Forest Products Industry [RISI] 2009). There is still room to improve the dimensional stability and durability of the panels in service. Lignocellulosic-based composites are liable to swell when they absorb water or moisture because of the hydrophilic nature of wood. Hemicellulose is mainly responsible for moisture absorption because of its available hydroxyl groups and also serves as a source of food for fungal decay (Das et al. 1999, Okino et al. 2007). The dimensional instability of OSB, especially in the form of thickness swelling (TS), not only creates aesthetic problems

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in some applications but also is associated with a loss in strength and stiffness of the material. The hydrogen bonds between hydroxyl groups allow microorganisms to enter the structure, interact with the polymers, and alter their properties (Papadopoulos 2006). OSB can be treated with toxic preservatives to prevent fungal attack (Fogel and Lloyd 2002, Smith and Wu 2005), but the treatment will result in indoor air-quality issues. Most of the OSB on the market is directly used in the building industry without any fungal-resistance treatment. Moisture plays a key role in the fungal degradation (Ye et al. 2006); improving moisture resistance can increase the durability as well (Hartley et al. 2007).

Heat and steam treatments have been used to improve the durability and dimensional stability of wood and wood-based composites (Tjeerdsma and Militz 2005, Okino et al. 2007). Heat and steam treatments decrease the number of free hydroxyl groups by removal of hemicellulose, decrease the hygroscopicity, and improve the dimensional stability and resistance to decay (Tjeerdsma and Militz 2005). For OSB, heat treatment can be applied either as a pretreatment of strands or as a post–thermal treatment of panels (Paul et al 2006, Okino et al. 2007). However, a drawback of heat treatment is that panel strength is reduced and an unpleasant odor is created.

The challenge is to improve the dimensional stability and durability of OSB without compromising its mechanical properties, using toxic chemicals, or increasing the cost of treatment. Extraction of hemicellulose from wood strands, in addition to providing sugars for the biofuel industry, may be a viable approach to increase dimensional stability and durability of OSB.

Extraction of hemicellulose sugars from wood strands by hot water has been investigated (Sattler et al. 2008, Paredes et al. 2009), but no significant enhancement in the properties of the panels has been demonstrated (Paredes et al. 2008). In our previous research, hemicellulose extraction of wood strands led to a decrease in hydrophilic character and water uptake of wood strands (Hosseinaei et al. 2011; Zhang et al., in press). After extraction, strand surfaces were covered with lignin components, which are less hydrophilic (Hosseinaei et al. 2011). Using the strands with a less hydrophilic character potentially can be helpful to improve dimensional stability and durability of OSB.

The objective of this study was to improve properties of flakeboard by extracting hemicellulose. The physical and mechanical properties of the flakeboards, as well as its susceptibility to mold, were studied.

Materials and Methods

Materials

Southern yellow pine (*Pinus* spp.) logs were obtained from J. M. Huber Corporation (Spring City, Tennessee). The logs were debarked and shipped to Louisiana-Pacific Co. R&D Technology Center (Franklin, Tennessee) for flaking. The average length of flakes was 15 cm, with a thickness of approximately 0.08 cm and random width. Fresh-cut flakes were directly shipped to North Carolina State University (Raleigh) for hemicellulose extraction.

Hemicellulose extraction

The hot-water extraction of hemicellulose was performed by a batch reactor with a total capacity of 70 liters. Treatment was performed at two different times (30 and 60 min) and three different temperatures (140°C, 155°C, and 160°C), with two repetitions for each condition. In each run, 5 kg of flakes, with an approximate moisture content (MC) of 70 percent, was loaded into the reactor at a water-to-solid ratio of around 20:1 (wt/wt). For exact measurement of the mass loss of the flakes due to the extraction, samples from 50 g of ovendried flakes (MC = 0%) were prepared and placed separately in a bandage cloth between the other flakes in the reactor. Mass loss of each sample due to extraction was calculated according to the following formula:

$$\mathrm{ML} = \frac{m_0 - m_1}{m_0} \times 100 \quad (1)$$

where ML is the percentage of mass loss, m_0 is the initial ovendried weight of the sample before extraction, and m_1 is the ovendried weight of the same sample after extraction.

After the reactor was loaded and the selected pretreatment temperature set, the reactor was heated at a rate of about 5°C/min. After the target temperature was reached, the temperature was maintained for the duration of the pretreatment. At the end of the process, the heat was disconnected, and the pressure of the reactor was gradually released to reach ambient pressure at a rate between 11.7 and 23.4 KPa/min, depending on the selected temperature. The severity factor (S_0) of the treatments was calculated from the following equation (Overend et al. 1987):

$$S_0 = \log\left\{\exp[\frac{T_\mathrm{r} - T_\mathrm{b}}{14.75}] \times t\right\} \quad (2)$$

where T_r is the temperature of the treatment (°C), T_b is the base temperature (100°C,) and t is the residence time (min).

To neutralize and clean the flake surfaces of organic acids, the flakes were soaked in NaOH solution (0.00036 N, pH 10) for 30 minutes and then rinsed with water until reaching about pH 7. Finally, the flakes were air dried and then oven dried at 70°C for 48 hours (MC ≈ 4%).

X-ray diffraction

To investigate the effect of extraction on the crystallinity of the wood flakes, x-ray diffraction (XRD) analysis was conducted with a Philips X'Pert materials research diffractometer using a Cu Kα radiation source. The XRD pattern was recorded within an angle range of 2θ from 5° to 60°, with a scanning rate of 0.04°/s. Wood flour was prepared by grinding the flakes (control sample and treatments for 60 min). Discs with a diameter of 13 mm and a thickness of 1.6 mm were made by compressing 0.2 g of wood flour. The relative degree of crystallinity (X_c) was calculated based on Segal method from the following equation (Segal et al. 1959):

$$X_\mathrm{c} = \frac{(I_{002} - I_\mathrm{AM})}{I_{002}} \times 100 \quad (3)$$

where I_{002} is the intensity of peak at 2θ = 22.7°, which represents both crystalline and amorphous parts, and I_AM is the intensity of diffraction at 2θ = 18°, which represents only the amorphous part.

Panel production

The pretreated flakes and a nontreated flake (as a reference) were then used to produce flakeboard. The flakes

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were blended with 5 percent liquid phenol-formaldehyde resin (Arclin, Inc., Dodson, Louisiana) and 1 percent wax emulsion (Hexion, Inc., Columbus, Ohio), based on the dry weight of the flakes. The mat was formed manually with random orientation of the flakes to a mat size of 18 by 18 inches (45.7 by 45.7 cm). Two panels were made for each extraction condition. The mat was pressed in a laboratory press at 200°C for 5 minutes to a panel with a thickness of 7/16 inch (1.11 cm) and a target density of 673 kg/m^3 (42 lb/ft^3).

Physical and mechanical test of panels

The panels were tested for their physical and mechanical properties. The modulus of rupture (MOR), modulus of elasticity (MOE), internal bonding (IB), water absorption (WA), and thickness swelling (TS) were tested according to ASTM D-1037 (ASTM International 2006). Cutting pattern of panels is presented in Figure 1. In addition, the equilibrium moisture content (EMC) of the panels was tested at 20°C and 65 percent relative humidity (RH). Samples with dimension of 1 by 3 inches were cut from bending samples, weighed, and placed in a conditioned room (65% RH, 20°C) until reaching constant weight (five replicates). After reaching the constant weight, samples oven dried at 103°C for 24 hours, and EMCs were determined based on the ovendry weight.

The data were analyzed with one-way analysis of variance (ANOVA) followed by Duncan's multiple range test for comparing means to determine the significance of variation. The ANOVA was performed with SPSS software, and the probability was set at 0.05 ($P < 0.05$).

Mold susceptibility test

The mold susceptibility of the test panels was evaluated using the method described in American Wood Protection Association (AWPA) E24 Standard Method of Evaluating the Resistance of Wood Product Surfaces to Mold Growth (AWPA 2006). Test samples, with dimensions of 3 by 2 inches, were cut from bending samples (six replicates). Samples were placed in a controlled-environment mold growth chamber, which was maintained at 25°C and near 100% RH. The samples were hung from stainless steel rods spanning the width of the tank. The artificial inoculums contained the following fungi: *Alternaria tenuissima, Aspergillus niger, Auerobasidium pullulans, Penicillium citrinum,* and *Trichoderma* spp. After 2, 4, 6, and 8 weeks of exposure, the samples were visually rated for the extent and intensity of mold growth on a scale of 0 to 5, where 0 indicates no visible mold growth and 5 is 100% mold coverage of the sample or colored growth obscuring more than 70% of the sample color. The MC of the samples was measured at the end of the test (8 wk) as well.

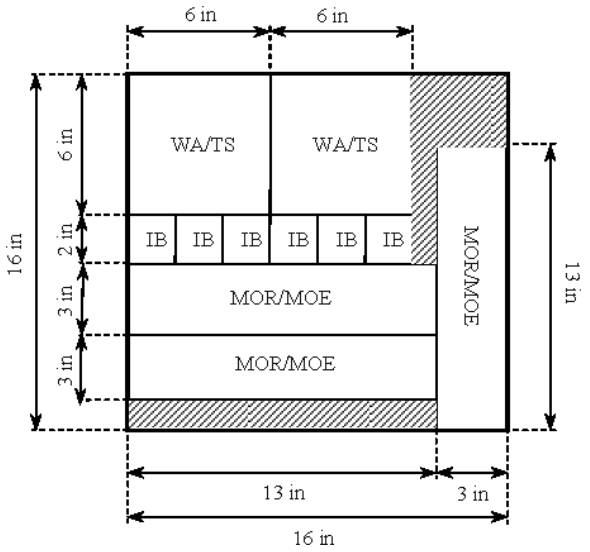

Figure 1.—Cutting pattern of the panels.

Results and Discussion

Mass loss of flakes after extraction

The influence of the extraction temperature and duration on mass loss is shown in Table 1. Mass loss increased with increasing severity to a maximum of 24.6 percent for the most severe condition (170°C, 60 min), which is in agreement with reported data for fractionation of lignocellulosic materials (Allen et al. 2001) as well as hemicellulose extraction (Sattler et al. 2008, Paredes et al. 2009). Hydrolysis of hemicellulose is the main reason for mass loss in flakes. Water under pressure penetrates the cell structure of wood, hydrates, and removes hemicellulose (Wyman et al. 2005). As pressure increases with temperature, this might be another reason for increasing mass loss with increasing temperature of treatment. Increasing the duration of treatment will result in increased production of organic acids, which catalyze the hydrolysis of hemicellulose and contribute to mass loss (Hosseinaei et al. 2011).

Effects of hemicellulose extraction on crystallinity of wood flakes

The crystallinity index of samples (Table 2) shows increase in crystallinity with removal of hemicellulose. A statistically significant difference is found between crystallinity of the control sample and the treated samples, and the highest crystallinity is observed for sample treatment at 170°C for 60 minutes. Increase in crystallinity after heat treatment of wood under highly moist conditions, which is similar to hot-water treatment, has been reported previously (Dwianto et al. 1996, Bhuiyan et al. 2000). Dwianto et al. (1996) referred to the degradation of hemicellulose, cross-linking reactions in the matrix substance, and crystallization of microfibrils during steam treatment of wood. Bhuiyan et al. (2000) mentioned that decomposition of the amorphous region and crystallization of quasicrystalline regions due to rearrangement or reorientation of cellulose molecules in these areas are the reasons for the increase in crystallinity of wood after heat treatment in highly moist conditions. Here,

Table 1.—Mass loss after extraction.

Temperature (°C)	Time (min)	Pressure (KPa)	Severity factor	Mass loss (%)[a]
140	30	283	2.66	6.4 ± 0.50
140	60	283	2.96	8.2 ± 0.24
155	30	483	3.10	12.8 ± 4.67
155	60	483	3.40	17.7 ± 5.08
170	30	717	3.54	19.9 ± 0.15
170	60	717	3.84	24.6 ± 3.08

[a] Values are means ± standard deviations.

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Table 2.—Degree of crystallinity of wood flakes.

Wood flakes		
Temperature (°C)	Time (min)	X_c (%)[a]
Control		56.8 ± 1.14 A
140	60	63.6 ± 0.14 B
155	60	65.7 ± 2.92 BC
170	60	67.6 ± 2.14 C

[a] Values are means ± standard deviations. Different letters indicate a statistical difference ($P < 0.05$) among the samples.

it seems that removal of hemicellulose, possibly some amorphous cellulose, and even some part of lignin increases the ratio of crystalline part to amorphous part and is the main reason for the increased crystallinity. Crystallization of amorphous regions needs a longer time or a higher temperature than used in this research (Dwianto et al. 1996, Bhuiyan et al. 2000).

Bending properties

The bending properties of panels (MOE and MOR) improved after extraction of hemicellulose and, under all the treatment conditions, were higher than in the control samples (Table 3). The statistical analysis indicates no significant difference in MOE among the different panels but a significant difference in MOR of the panels (Table 4). Panels made from treated flakes had much higher MOR than panels made from untreated flake. The panel MOR decreased as the severity factor increased except for the panel treated at 155°C for 60 minutes. This panel had the highest density (716 kg/m^3), which could be one of reasons that this panel was not fitted with the trend. Further research is needed.

The increase in the bending properties might be attributed to the increase in the ratio of cellulose and the crystallinity of wood flakes after treatment. XRD analysis has proved the increase in crystallinity of wood after hot-water treatment (Table 2). Because of its high degree of polymerization and crystallinity, cellulose is responsible for the strength in wood fiber (Yildiz and Gumushkaya 2007). According to the statistical analysis, panels from the less severe treatment show higher bending properties compared with those from the most severe treatment. This may be because of possible damage to the middle lamella, which bonds the fibers together, due to possible hydrolysis or migration of the lignin.

Another reason for the improvement in the bending properties might be that the density of wood flakes decreases after extraction. Panels were made from extracted flakes using a greater volume of flakes compared with the control panels, which were made from control flakes. A decrease in the density of flakes increases the compressibility of flakes during hot pressing and helps improve bending properties.

Internal bonding

The IB of the panels is shown in Table 3. The extraction treatment resulted in a decrease under some conditions, especially for panels containing wood treated at 170°C; these IB values were significantly lower than those of other panels (Table 4). Although IB decreased in some cases, all the IB values met the IB requirements of the Canadian Standards Association (Canadian Standards Association 2005).

Strong adhesion between the adhesive and wood is achieved by appropriate adhesive flow, penetration, wetting, and curing (Sernek et al. 2008). Extraction treatment resulted in a decrease in the hygroscopicity of the flakes; this can alter the distribution of the adhesive on the wood surface. The intensity of water absorption from the waterborne adhesive can affect the hardening process of the adhesive and, consequently, the quality of the adhesive bond (Sernek et al. 2008). Studies on heat treatment of wood have shown that wettability of wood decreases after heat treatment and that the surface of heat-treated wood is hydrophobic, less polar, and significantly repellent to water (Hakkou et al. 2005). Measurements of the contact angle and surface free energy before and after extraction indicate a significant decrease in the hydrophilicity of flakes after extraction (Hosseinaei et al. 2011), which can negatively affect the distribution and penetration of the waterborne resins that are used for making panels.

Possible degradation of lignin under high severity factor conditions can damage the middle lamella and affect the bonding of fibers, also negatively affecting the results of the IB test. Another factor that could affect the bonding process is pH. Producing organic acids during hemicellulose extraction resulted in a decrease in pH (Hosseinaei et al. 2011). Although the flakes were neutralized and washed after extraction, it is possible that the pH value of the treated flakes was still less than that of the untreated flakes and may have affected the curing of phenol-formaldehyde resin.

Table 3.—Mechanical and physical properties of flakeboards[a]

Extraction conditions		Density (kg/m^3)	MOE (MPa)		MOR (MPa)		IB (MPa)		EMC (%)[b]	
Temperature (°C)	Time (min)	Mean ± SD	Mean ± SD	CV	Mean ± SD	CV	Mean ± SD	CV	Mean ± SD	CV
Control		687 ± 8.6	5,360 ± 940	17.5	35.5 ± 4.82	13.6	0.56 ± 0.12	22.1	9.07 ± 0.35	3.85
140	30	709 ± 7.2	6,670 ± 1,350	20.2	52.7 ± 13.1	24.8	0.53 ± 0.08	15.1	8.73 ± 0.28	3.18
140	60	700 ± 3.5	5,950 ± 1,200	20.1	50.2 ± 12.1	24.7	0.44 ± 0.07	15.2	8.95 ± 0.25	2.79
155	30	708 ± 5.7	6,300 ± 1,920	30.4	45.5 ± 11.1	24.3	0.56 ± 0.12	22.1	7.90 ± 0.22	2.74
155	60	716 ± 2.7	6,310 ± 1,090	17.2	52.2 ± 4.05	7.76	0.59 ± 0.12	20.5	7.90 ± 0.43	5.45
170	30	706 ± 2.1	5,640 ± 818	14.5	40.5 ± 6.57	16.2	0.34 ± 0.02	6.14	7.54 ± 0.12	1.56
170	60	695 ± 8.3	5,780 ± 1,090	18.8	39.3 ± 9.26	23.6	0.34 ± 0.05	16.1	7.01 ± 0.10	1.38

[a] MOE = modulus of elasticity; MOR = modulus of rupture; IB = internal bond; EMC = equilibrium moisture content; SD = standard deviation; CV = coefficients of variation.
[b] EMC at 20°C and 65 percent RH.

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Table 4.—One-way ANOVA followed by Duncan's multiple range test.[a]

Extraction conditions							
Temperature (°C)	Time (min)	MOE	MOR	IB	WA	TS	EMC
Control		—	A	BC	C	D	D
140	30	—	D	BC	B	C	D
140	60	—	BCD	B	B	C	D
155	30	—	ABCD	C	B	AB	C
155	60	—	CD	C	B	C	C
170	30	—	ABC	A	AB	BC	B
170	60	—	AB	A	A	A	A

[a] MOE = modulus of elasticity; MOR = modulus of rupture; IB = internal bond; WA = water absorption; TS = thickness swelling; EMC = equilibrium moisture content. Different letters indicate a statistical difference ($P < 0.05$) among different conditions.

WA and TS

Both WA and TS were significantly reduced by the extraction process and the extraction of hemicellulose improved the dimensional stability of panels (Fig. 2). The lowest WA and TS were observed in extraction at 170°C for 60 minutes, conditions that presented about 52 and 38 percent reduction, respectively, compared with control panels. Panels made from untreated flakes showed the highest amounts of both WA and TS and were significantly different from those of panels made with any of the extracted samples (Table 4).

Studies of steam treatment and heat treatment of wood also show a decrease in hygroscopicity and EMC and an improvement in the dimensional stability of wood (Hsu et al. 1988, Das et al. 1999, Paul et al. 2006). Hygroscopicity is highly correlated with the accessible hydroxyl groups in wood (Tjeerdsma and Militz 2005), so we assume that the decrease in the hygroscopicity of wood flakes after extraction is due to the reduction of accessible hydroxyl groups of wood. Das et al. (1999) have stated that hemicellulose is mainly responsible for moisture absorption, but the accessible cellulose, noncrystalline cellulose, and lignin also play major roles. As reported in an earlier study (Hosseinaei et al. 2011), the surfaces of flakes after hemicellulose extraction demonstrate a decrease in hygroscopicity. Hot-water treatment also has resulted in decreased water uptake of wood strands (Zhang et al., in press).

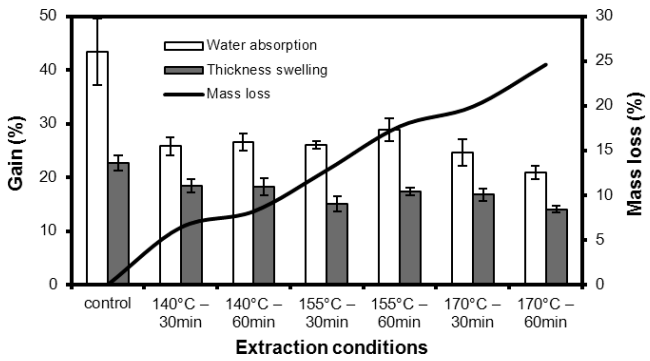

Figure 2.—Water absorption and thickness swelling of samples and mass loss of flakes due to extraction.

Removing hemicellulose is probably the main reason for the reduction of the hydroxyl groups and the hygroscopicity. Tjeerdsma and Militz (2005) noted that the reduction of accessible hydroxyl groups in hydrothermal treatment of wood can also be explained by the occurrence of cross-linking reactions or by recrystallization of the cellulose. Lignin deposits on the surface of flakes after extraction (Hosseinaei et al. 2011) may also contribute to decreased hygroscopicity of wood flakes. The increase in the contact angle of water on flakes and the decrease in the surface free energy after extraction of hemicelluloses proved that extracted flakes are less hydrophilic in character (Hosseinaei et al. 2011).

Equilibrium moisture content

The EMC of panels that occurred at 20°C and 65% RH is given in Table 3. As expected from the WA results, the EMC of boards made from extracted flakes was lower than that of the control panel, and the EMC decreased in severe conditions. Statistical analysis shows a significant difference at the 95 percent confidence level between the EMC values of different conditions (Table 4). The lowest EMC was obtained for the extraction at 170°C for 60 minutes and showed a 23 percent reduction compared with the EMC of the control board. The decrease in EMC shows that after extraction, the hygroscopicity of the panels decreased; this is related to the decrease in accessible hydroxyl groups and the covering of the surface of the flakes with hydrophobic materials, as explained for WA. A statistically significant difference was not found between the control sample and the samples that were extracted at 140°C (30 and 60 min), because most of the hemicellulose still remained in the wood after extraction.

Mold susceptibility of flakeboards

The flakeboards made with the extracted flakes in all conditions were more resistant to mold than panels made with untreated flakes or the solid wood (pine) controls (Fig. 3). A trend toward increasing mold resistance with increasing severity factor of the reaction was observed.

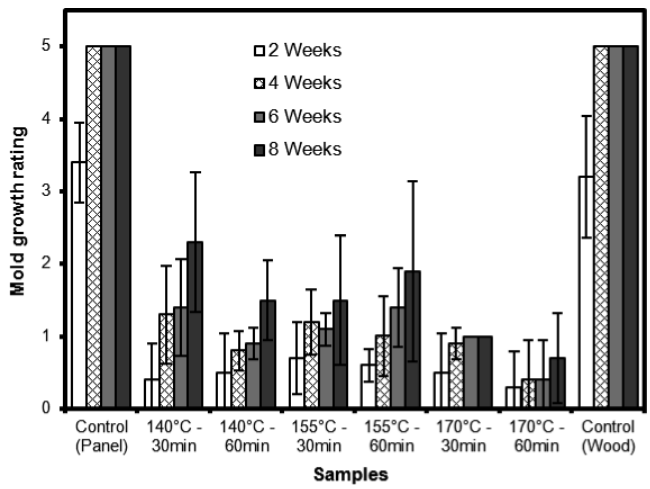

Figure 3.—Mold rating of flakeboard and solid wood samples.

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The extraction removed a significant amount of materials (Table 1), some of which may have served as food for the mold fungi. The production of degradation products during treatment may have been another reason that the fungal activity was affected (Hosseinaei et al. 2011). The treatments also altered the hygroscopicity of the wood (Fig. 2) and, thus, may have indirectly affected the susceptibility of the wood to fungal attack. However, the MC of the exposed samples was measured at the end of the test (8 wk of exposure) and in all cases, the average MC of the samples was high enough to support fungal growth (Fig. 4).

These data suggest that the extraction of hemicellulose from wood flakes before flakeboard manufacture can result in panels with increased resistance to mold growth. This resistance may be due to a combination of the removal of potential food sources important to the fungi and the production of inhibitory compounds.

Conclusions

Extraction of hemicellulose from wood flakes decreased the hygroscopicity of flakeboards, and the panels with a less hygroscopic character showed better dimensional stability and mold resistance. The highest mass loss of flakes due to extraction treatment results in the lowest WA and TS of panels; the mold resistance of panels made using the most severe condition was the highest. Extraction of hemicellulose offers the potential for producing both bioproducts/biofuels and improving the durability and dimensional stability of building materials without decrease in mechanical properties and without using toxic chemicals.

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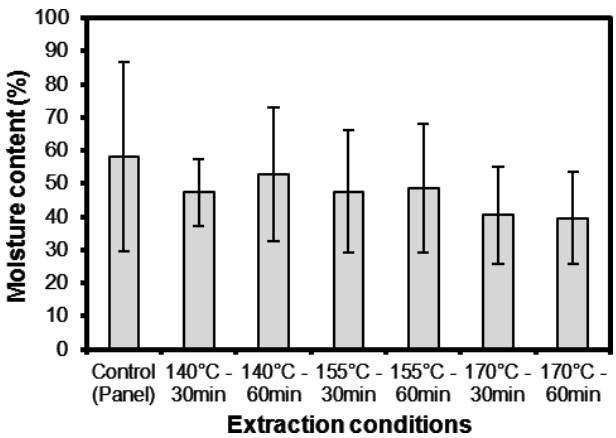

Figure 4.—Moisture content of the samples after 8 weeks of exposure to mold.

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