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ORIENTED STRAND BOARD (OSB) FROM HOT WATER EXTRACTED WOOD

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SUMMARY

The increased cost of petroleum and policy changes due to concerns about climate change are game changing events which will create major changes in the economy. One concept that has received attention is that of a forest bio-refinery, wherein multiple products are produced. While integrated forest products complexes have been the norm for many years, the difference in biorefinery is the emphasis on chemical and materials production which compete with items traditionally manufactured from petroleum. There are several sulfite pulp mills in the world which produce a variety of chemicals in conjunction with the manufacture of pulp. In the United States, Red Shield Environmental is evaluating the potential for co-production of acetic acid and ethanol from a kraft mill facility. This concept, wherein multiple material and chemical products are produced from wood, can be applied to other forest products production scenarios. The Oriented Strand Board composite industry is a good candidate due to large plant sizes as well as technical considerations including rapid mass transfer rates due to the thinness of the wood. The production of OSB in North America for 2006 was 14.24 million tones. A hot water extraction process primarily removes hemicellulose compounds. Assuming a 15% weight removal with subsequent conversions could result in an annual production of 5.19 million barrels of ethanol. Assuming a market for such an extract, the question becomes what is the influence of the extraction on the behavior of the core high-value forest product?
This research was conducted using three Red Maple (***Acer Rubrum*** L.) trees, three strand thickness (0.025", 0.035", and 0.045"), and two hot water extraction time of 45 and 90 min. at 160 C and control (unextracted). One OSB panel was manufactured for each of the 27 material combinations. Weight loss was significantly influenced by extraction time and tree source. The thickness swell and sorption isotherms of panels from extracted material was lower while its water absorption was significantly higher than the control. Fungal deterioration was similar for control and extracted material. The higher heating value was increased after hot water extraction. Modulus of rupture (MOR) was not statistically significant different between the unextracted and 45 minute extraction conditions but it was decreased after 90 minutes. Internal bond for both extraction time (wet and dry conditions) was significantly lower than the control. The distribution and size of pores within the cell wall increased as the extraction time increased. Contact angle indicated more pronounced liquid wetting and penetration for the extracted material. Inverse gas chromatographic identified that dispersive surface free energy and acid characteristics increases with extraction time. The percentage of crystallinity of cellulose increased as a function of the extraction time.

INTRODUCTION

In the 150 years since the first oil well was drilled in 1859 in Titusville, Pennsylvania, there has been a fundamental shift toward the use of petroleum for energy and materials for the world economy. Recent rapid increases in the price of petroleum along with the confluence of carbon emission influences on climate change has created fundamental economic and policy shifts toward sustainable and renewable sources of materials and energy. This has renewed interest in the ability to provide a wide range of products (materials, chemicals, and energy) from sustainably managed forests and other plant materials.

The current global forest products industry is one primarily focused on the production of materials (e.g. lumber, pulp, panel products) and energy (typically for use within the manufacturing plant). Its use as a chemical feedstock is more limited. Historically, the forest provided many important industrial chemicals (methanol, tannic acid) but these products were displaced by cheaper petroleum derived alternatives.

Under the assumption that a fundamental market shift is occurring, this research was conducted to evaluate the appropriateness of simple water extraction to create a chemical feedstock stream within the context of Oriented Strand Board (OSB) manufacture.

BACKGROUND

The simplest process for the removal of chemicals from wood is hot water extraction. The temperature and time associated with the extraction can create a wide range of chemical reactions and modifications of the wood. Hydrothermal condition result in wood weight loss and the removal of sugars (Garrote et al., 1999). Heating in the water of wood result in the formation of organic acids (generally acetic acid) that catalyze the hydrolysis of hemicelluloses, while the amorphous cellulose are less affected (Mitchell, 1988 and Garrote et al., 1999). The remaining hemicelluloses are degraded more than other macromolecules compounds (Jara et al., 2006 and Hill, 2006) and the degradation of the cellulose and lignin can become significant above 180 °C. The attainment of temperatures above 100 °C requires a pressurized environment.

Within the temperature range of 140 °C to 230 °C, lower temperature produce very slow hydrolysis and above 210 °C become the cellulose degradation (Yoon et al., 2006 and Hill, 2006). Hemicellulose can be removed at temperatures as low as 100 °C (Ramiah, 1970). Hardwoods are less thermally stable than softwood due to hemicelluloses content and composition (Hill, 2006). Xylans (pentoses) had been found in higher percent in hardwood species and galactoglucomanns (hexosans) had been in higher percent in softwood (Fengel and Wegener, 1989). Hardwoods also have a higher content of hemicelluloses and a higher acetyl proportion than the softwoods, thus hardwood have the highest rate of mass loss (weight loss) in similar hydrothermal conditions (Hill, 2006).

Cellulose degradation occurs at a higher temperature than hemicelluloses, beginning in the amorphous regions, but if water is present, these amorphous portions of cellulose can change its structure to produce more thermally stable crystalline regions (Fengel and Wegener, 1989). It is generally accepted that lignin is the most thermally stable component of the cell wall. Under pressurized steam conditions with temperatures at 180 °C it was found that there was a slightly modified syringyl groups of the lignin, but at higher temperatures and longer times of exposure the lignin was susceptible to degradation (Sudo et al., 1985; Nuopponen et al., 2004). Therefore, the extraction time and temperature are important process variables which impact the amount of material removed from the cell wall as well as the extent of change in cell wall structure and chemistry through autohydrolysis.

The kinetics of removal of cell wall material by chemical processes has been extensively studied in the pulping literature is beyond the scope of this work. However, the kinetics are typically strongly correlated with diffusion processes. As such, the thinness of OSB wood strands (0.025 to 0.055 inch) reduces the process times needed.

Several wood modification methods had been investigated and developed commercially to improve the wood properties (Hill, 2006). One of them is hydrothermal treatments to improve the dimensional stability of solid wood. The resultant product does not require coatings and exhibits a degree of resistance to biological attack of the lumber (Dirol and Guyonnet, 1993; Troya and Navarette, 1994; Howell et al., 2008). Hydrothermal treatment had been used as commercial process in Finland with an increase in modified lumber production increased from 50,000 m^3 to 80,000 m^3 over the years 2000 to 2005 (Hill, 2006).

The influence of the extraction on the wood properties will have a subsequent influence on that of the OSB produced from the extracted wood. Hypothesized benefits of producing OSB from the chemically modified wood include the production of lower density composite material, reduced VOC production during drying and pressing, and improved dimensional stability. However, excessive degradation of the woody material due to hydrolysis could impact gluability and result in low mechanical performance.

METHODOLOGY

Extraction

Three Red Maple (*Acer rubrum* L.) trees were felled and debarked. The butt log was used to create 10 cm long strands at target thickness of 0.64, 0.89 and 1.14 mm using a 12/48 Ring Carmanah Strander. The strands (500 g) were placed inside a high pressure vessel (digester) filled with sufficient fresh water (tap water) a liquid-solid weight ratio of 4. The two extraction protocols involved heating from room temperature to 160 °C in 50 min. (preheating time) followed by constant temperature exposure times of 45 or 90 minutes.

These two extraction conditions were equated to a severity factor through the use of Equation [1] (Overend and Chornet, 1987; Mosier et al., 2002).

$$SF = \log\left(\int_0^t Exp\left[\left(\frac{T_r - T_b}{14.75}\right)\right]dt\right)$$

Eq. 1

Where, t is the residence time (min.), T_r is the reaction temperature (°C), and T_b is the base temperature at 100 °C. The energy of activation is 14.75 (kJ/kg-mol) assuming that the overall process is hydrolytic and the overall conversion is first order (Overend and Chornet, 1987; Jeoh, 1998; Mosier et al., 2002).

Both prior to and following extraction of the wood strands, the temperature of the water was measured at the heater element at three second intervals. Using Equation [1] a SF of 3.54 and 3.81 was calculated for the 45 and 90 minutes extraction times, respectively. Strand weight loss as a result of the extraction process was determined for each extraction run by freeze drying the extracted liquid, temperature -42°C and between 13 and 15 x 10^{-3} Mbar vacuum (Paredes et al., 2008a).

OSB production and testing

Strands were conditioned at 37.8 °C and a RH of 60% for unextracted (control) material and a RH of 80% for hot water extracted material for five days until constant weight was attained, resulting an equilibrium moisture content (EMC) of the extracted strands was significantly lower than the control strands (8.1 vs. 10.4%), even though the extracted strands were exposed to a higher RH.

One OSB panel (44.7 x 31.4 x 1.3 cm) was manufactured for each of the 27 material combinations (2 extraction conditions and unextracted, 3 trees, and 3 strand thickness). Panels were blended individually with a resin load of 3.2% pMDI resin, no wax, and an average density of 41.8 pcf (test volume basis). Manufacturing conditions of the OSB can be found in Paredes et al (2008a), physical and mechanical properties of that panels were determined following ASTM D1037-06A procedures. Treated and untreated boards were tested for decay susceptibility in a modified ASTM soil block jar bioassay using the white rot fungus *Pycnoporus sanguineus* and the brown rot fungus *Meruliporia incrassata* (Howell et al., 2008).

Wood evaluation

The surface and cross section of selected wood strands were evaluated with the use of a scanning electron microscopy (SEM), the distribution and size of the cell wall pore structure was determined by mercury porosimetry (ISO/AWI 15901-1), the contact angle of distilled water, diiodemethane, and ethylene glycol on specimens was determined by the sessile drop method, and inverse gas chromatographic (IGC) was used to identify dispersive surface free energy and acid base characteristics (Paredes et al., 2008b). The degree of crystallinity and width of crystal for control and two hot water extractions were scanned using a Panalytical X'Pert XRD machine with symmetric θ-2θ Bragg-Brentano scattering geometry (Howell et al., 2006; Paredes et al., 2008b).

High heating values (HHV) were determined for each severity factor (SF) and control from strand thickness of 0.035". The OBS strand samples were chopped using a Wiley Mill process with a 0.5 mm mesh screen. The ground fibers were then dried in lab oven chamber at 103 C for 24 hours.

Specimens form the liquid extracted of the strands as a result of the extraction process were chosen from the same tree and strand thickness used in the OSB strands at a SF 3.54. The dried fibers (0% MC) were pressed into calorimeter samples using a Parr 2881 pellet press and each sample weighed around 0.5 g. Then every pellet was placed in the bomb (combustion capsule). A fuse wire was placed between two electrodes while at the same time touching the pot of the pellet. This ensured that the pellet would be burnt upon ignition of the fuse wire (PARR 45C10 nickel-chromium). The bomb was charged with 4 MPa of oxygen.

Heat of the combustions were measured using a Parr Model 1241 adiabatic calorimeter. To calculate GHV of combustion in calories per gram by substitution in the Eq. 2 (Rice and Willey, 1995).

$$GHV = \frac{tW - e_1 - e_2 - e_3}{m}$$

Eq. 2

Where t is the temperature rise $(t = t_f - t_a)$, t_a is temperature at the time of firing, t_f is final maximum temperature, e_1 is correction in calories of heat of formation of Nitric acid (HNO_3), e_2 is correction in calories for heat of formation of sulphuric acid (H_2NO_3), that was assumed zero for this laboratory, e_3 is correction in calories for heat of combustion of fuse wire, m is mass of specimen in grams, and W is the energy equivalent of calorimeter in calories per degree Celsius.

RESULTS AND DISCUSSION

Weight loss

During hydrothermal treatment, wood experiences a decrease in mass, volume, bound water and extractives volatiles, which is dependent of temperature, time of extraction, species, material dimensions, etc. (Mitchell et al., 1953; Seborg et al., 1953; Stamm, 1956; Jara et al., 2006; Paredes et al., 2008a). Paredes et al. (2008a) found that the weight loss for Red Maple (*Acer rubrum* L.) was significantly influenced by time of exposure ($p = 0.0011$) of 45 and 90 min. plus 50 min. of preheating (Figure 1), resulting in a mass loss of 16.4 ± 0.3% and 17.2 ± 0.3%, respectively. Tree source also had a significant effect ($p = 0.0001$), while strand thickness of 0.025", 0.035", and 0.045" was not statistically different ($p = 0.1026$).

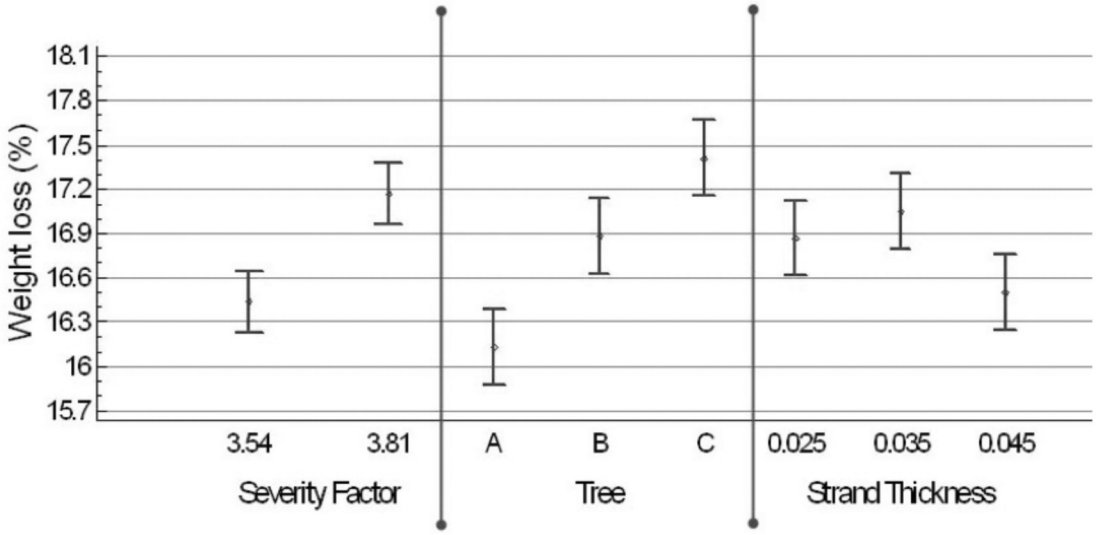

Figure 1: Effect of severity factor (SF), tree source, and strand thickness on weight loss during hot water extraction at 160°C (Paredes et al., 2008a).

After hot water extraction, a darkening of wood occurs related to the severity factor. Chow and Mukai (1972) found that the color change had a relationship with hydrothermal conditions, degree of crystallinity, degree of polymerization and OH content.

OSB physical properties

The equilibrium moisture content (EMC) at 23 C/ 65% RH of the OSB panels was depressed from approximately 10 to 6%. This is consistent with the removal of hemicellulose and the increase in cellulose crystallinity associated with the pre-extraction process. Swelling of the OSB panels during the equilibration process was monitored (Figure 2) with the OSB manufactured from extracted strands exhibiting less thickness swell and a more rapid equilibrium conditioning time.

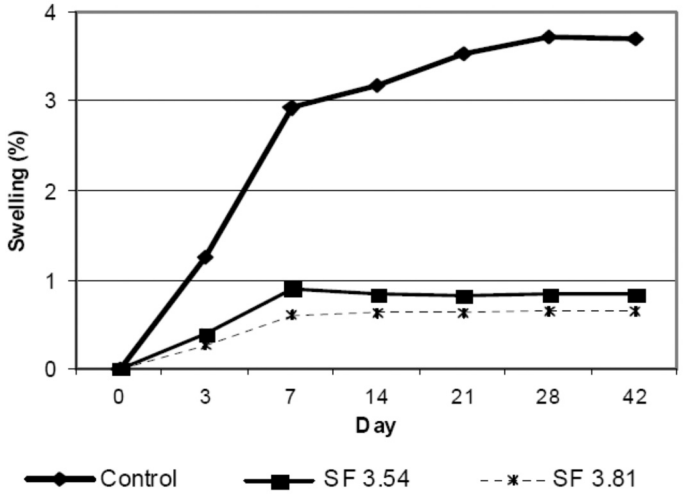

Figure 2: Panel thickness increase occurring from out of press to equilibrium conditions (23 C/ 65% RH).

Stamm et al. (1946) and Seborg et al. (1953) also found that the change in dimensional stability was dependent upon weight loss (%), temperature, time and atmosphere employed (air, nitrogen, etc.), reaching a maximum at about 20% weight loss.

Heat treatments of wood are widely used to reduce the wood swelling and to improve the dimensional stability (Stamm et al., 1946; Repellin and Guyonnet, 2005; and Hill, 2006). Interestingly, the lower EMC conditions of the OSB material did not fully translate to improved OSB dimensional stability during water immersion (Figure 3).

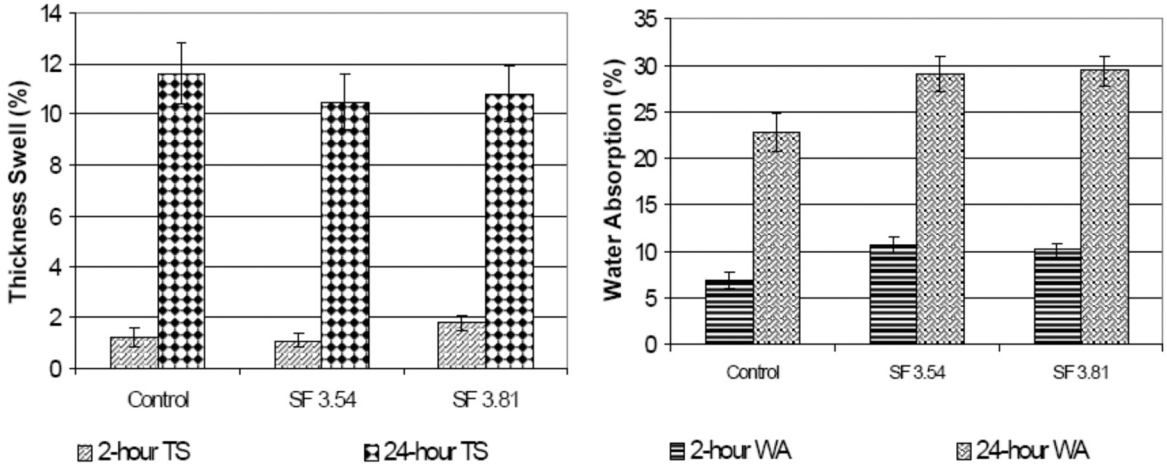

Figure 3. Thickness swell and water adsorption response of red maple control and hot water extracted panels OSB (Paredes et al., 2008a).

There was a statistically non-significant decrease in the 24-hour thickness swell due to the use of extracted wood strands. Thickness swell in OSB is influenced not only by the behavior of the wood (expected to be less for the extracted wood), but by adhesive bond behavior. Low internal bond (IB) properties of the extracted OSB (see following section) indicate these interacting factors may have resulted in the lack of thickness well improvement. Water absorption of the panels was significantly increased. Thickness swell and water absorption are typically highly correlated. This decoupling is likely due to the increased cell wall porosity from the \extraction as well as the lower starting EMC of the extraction OSB.

Previous studies have shown that extraction of hemicellulose from wood using high pressure steam caused a reduction the thickness swell in both hardwood siding and wood (Lenic, 1973; Myers, 1982; Spalt, 1988 cited by Carll 1997; Wang et al., 2000). Hsu (1988) reported that hydrolysis of hemicellulose by high pressure steam reduced the thickness swell of particleboard and waferboard. The reduction in hygroscopicity, capillary flow, and dimensional movement is due to process of replacing hydroxyl groups in lignocellulosic materials with acetyl groups (Carll 1997). Consequently, the actual fiber saturation point (FSP) is modified when the sugars are removed from the wood.

OSB mechanical properties

The effect of hydrothermal treatment from red maple OSB panel is shown in Figure 4.

Figure 4: Changes in bending modulus of rupture (MOR), bending stress at proportional limit (SPL), bending modulus of elasticity (MOE), and internal bond (IB) as a result of hot water extraction.

The bending strength (MOR) was similar for the control and short extraction time (SF 3.54) panels while there was a significant decrease in MOR for the longer extraction time. It has been shown that the bending strength of wood is reduced when heated in air, nitrogen or pure oxygen (Stamm, 1946; Mitchell, 1988) However, in moist conditions, the rate of decrease accelerates and does not obey first order kinetics (Hill, 2006). The stress at proportional limit (SPL) behavior of the red maple OSB panels exhibited the same trend as MOR, i.e. control and low severity factor (3.54) panels were similar, with a decrease in performance at the high severity factor (3.81). The slight increase of MOR after hydrothermal conditions also has been found under nitrogen or air treatments in short time periods at temperatures ranging from 115°C to 175°C in closed systems, but when the time was increased resulted in a reduction in MOE (Kubojima et al., 2000; Mitchell, 1988; Chang and Keith, 1978).

In contrast, the internal bond (IB) of the panels were significantly lowered at even the lower SF value. This was true for both wet and dry conditions. It may be that the modification of the wood strands results in over penetration of the adhesive, resulting in low wood/wood bond strength and hence lower IB (Paredes et al., 2008a).

OSB decay resistance

Numerous decay studies have demonstrated that thermally modified wood improves decay resistance (Stamm et al., 1946; Dirol and Guyonnet, 1993; Troya and Navarette, 1994). The protection against brown rot fungi and white rotters is expected due to loss of polysaccharides. Howell et al. (2008), using Red Maple OSB specimens from this study found that the decay susceptibility (weight loss) under a modified ASTM soil block jar bioassay was not statistically different for the brown rot fungus *Meruliporia incrassata*. However, weight loss was significantly lower in extract OSB in response to the white rot fungus *Pycnoporus sanguineus*.

It is known that the decay mechanism employed by brown rots are different than that of white rot fungi. White rot fungi generally attack the hemicelluloses and lignin while brown rot fungi metabolize the hemicelluloses (Zabel and Morrel, 1992). Chemical analysis of the liquid extract show it to primarily be composites of hemicellulose with a small amount of lignin and volatile extractives (Jara et al., 2006) While the preferential food of the brown rot fungus, cellulose, had not been removed by hot water process, it has also been reported that they have resistance against certain wood preservatives and other chemical (Zabel and Morrel, 1992; Goodell et al., 2002). Increases in the cell wall pore structure due to the extraction process (Paredes et al., 2008b) may also allow the *M. incrassata* to penetrate the cell wall and colonize easier (Howell el et., 2008).

Cell wall microstructural changes

Ultrastructural investigations indicated that the pressurized hydrothermal process did not induce cellular collapse but did show significant cell wall damage in the form of pitting, with the most pronounced damage occurred in long extraction time (Paredes et al., 2008b). Using mercury porosimetry, it was determined that pore diameter show increase in range from 300 to 600 nm in the extracted material compared to the control, and that there is an increase in intra cellwall pore structure of approximately 22.2% (Paredes et al., 2008a). Hietala et al. (2002) reported that when wood samples were thermally treated there is no change to the size or distribution of cell wall micropores at 115°C, but it was significantly increased when the temperature exceeded 180°C with an average size of these micropores of around ten nanometers determined by nuclear magnetic resonance (NMR) technique. Wang and Yan (2005), in resin penetration study, found that the bigger pores (diameter > 40 μ) were filled partially with phenol-formaldehyde resin while the pores with diameter > 1 μ were completely filled. The increased of porosity and good penetration of resin in the smallest pores of hot water extracted wood indicate a penetration of resin into wood result a weaker IB in the extracted material than in the control.

The supermolecular structure of wood cellulose crystallinity and crystallite width were determined by comparing the areas under crystalline peaks and the amorphous curve, using an x-ray diffraction pattern.

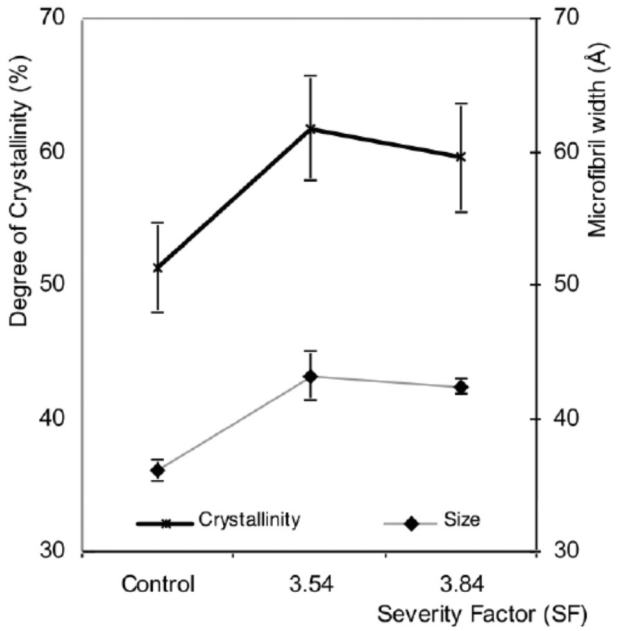

Figure 5: Crystallinity degree - microfibril width behavior of red maple control and two severity factor in hot water treatment at 160°C.

The changes in the supermolecular structure of the respective polymorph forms of cellulose for the control and two severity factor condition specimens were analyzed as a function of the pre-extraction conditions (Figure 5). The determined percent crystallinity values were 51.3 ± 3.3% for the control, 61.7 ± 3.9% for SF 3.54 and 59.5 3 ± 4.1% for the SF 3.81 treatment. The percentage of crystallinity was significantly influenced by pre-extraction conditions (p= 0.0001). Crystallite width varied in a similar manner as percent crystallinity. Similar trends have been observed in other wood species (Hirai et al. 1972; Bhuiyan et al. 2000; Akgül et al., 2007), hot water extracted wood of other species (Howell et al. 2008a) and wood degraded by fungi (Howell et al., 2008b). The changes in crystallinity were explained as crystallization in quasicrystalline or sub crystalline structures found by Ding and Himmel (2006), attributed to rearrangement or reorientation of cellulose molecules and hemicelluloses rather than loss of amorphous material (Bhuiyan et al., 2000). Increases in the crystallite width of extracted wood indicated a swelling process due to application of hot water. Sarko (1996) applied sodium (Na) to microfibrils of cellulose which resulted in an increase in width due to a swelling process. Also, a reorientation process (Hill, 2006) that can cause fiber aggregation through the removal of hemicelluloses may be taking place (Emons 1988; Jarkis 1990 cited by Ding and Himmel 2006).

Wood surface modification

The development of a good adhesive bond requires the spreading, wetting, and penetration of an adhesive into the adherent (substrate). A common measure of wetting is contact angle, which was established by work originally done by Young. Contact angle measurements indicate the wettability of a solid surface by a liquid.

The contact angle formed by all the liquids (distilled water, diiodemethane, and ethylene glycol) was less than 90°, therefore the liquids were able to penetrate the porous wood. In the extracted wood strands, the contact angle determined using diiodemethane, exhibited the fastest penetration into the strand surface. Complete penetration of the liquid into the wood occurred within a few seconds. The penetration of liquids into the wood structures is initialized by penetration of the gross capillary structure, lumens and intercellular pits, followed by movement into the spaces within the secondary wall. The more severe pre-extraction conditions (SF 3.54 and 3.81) were shown to have higher pore volume in the earlier mercury porosimetry results. This translates to increased permeability and is consistent with the more rapid liquid penetration of the material.

The process of manufacturing OSB depends on an good adhesive bond, typically using a polymeric diphenylmethane diisocyanate (pMDI) or phenol- formaldehyde (PF) resin system. Generally, pMDI represents as little as 2 percent of dry furnish weight (Kamke and Lee, 2007). The bondlines within OSB are discontinuous and the thickness is irregular because the OSB strands are not completely covered with resin (Conrad et al., 2004). The increased permeability of the pre-extracted material may have resulted in an over-penetration of resin in the extracted strands, thereby contributing to the reduction in internal bond (IB) reported by Paredes et al. (2008a). A common measure of wetting is contact angle, which was established by work originally done by Young. In the hot water treated strands, the contact angle with diiodemethane, distilled water, and ethylene glycol, had the fastest penetration of the liquids placed on their surface. The penetration time was only a few seconds, resulting in an over-penetration of the liquids in the strand surface (wood) microstructure.

The Schultz method was utilized for the determination of the dispersive surface energy of the maple samples. The results indicate that as more hemicellulose is removed from the surface, there is an increase in surface energy of the wood (Figure 6).

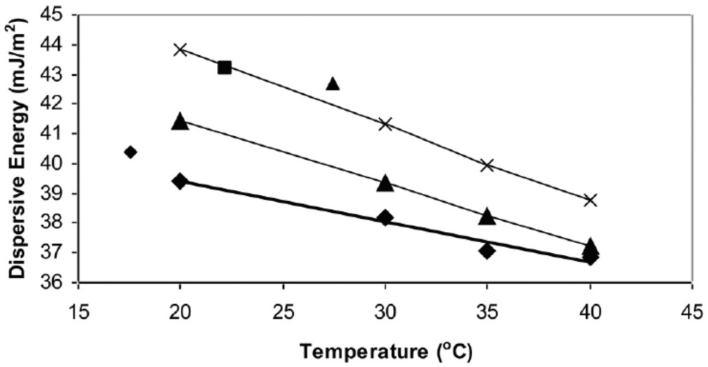

Figure 6. Dispersive surface energy calculated by the Schultz method for extracted maple residues, ♦ is control, ▲ is SF-3.54, and **x** is SF-3.81.

Heat treatment on the surface tends to reduce the wettability of the wood, especially at higher temperatures, which interferes with diffusion of glue moisture into the wood which often results in undercured bonds (Hancock, 1963).

Wood higher heating value

Since the material was at a 0% moisture content, the GHV and HHV values are identical and only the GHV values are reported. Table 1 outlines the results for the experimental variables.

Table 1: Influence of hot water extraction on gross heating values (GHV) of red maple.

Sources	Count	Moisture Content (%)	GHV (cal/g wood)	COV (%)	p-value
			Mean		
Control	4	0	4629.2	2.1	0.0000
Extracted Liquid Solids	2	0	4407.0	3.1	
SF 3.54	2	0	4698.7	2.9	
SF 3.81	2	0	4694.0	2.9	

There was a clear reduction in the heat content of the extract material (approximately 66 calories per gram of ovendry wood). This is consistent with a rule of mixtures analysis wherein the removal of lower heat content hemicelluloses (4407 cal/g) from the wood left the remaining wood with a higher relative lignin content and hence an overall increased energy content. The relative heat content of the extract is critical for evaluation of net process energy inputs and recovery for subsequent conversions to biofuels.

CONCLUSIONS

The implementation of biorefinery concept in the product of OSB was explored in which hot water extraction was used to remove a significant amount of the wood material for conversion to other products while assessing the impact of the extraction on the OSB performance. The extraction influence of fundamental wood properties was also investigated to help explain performance changes. The hot water extraction treatments which resulted in weight loss values of up to 17%. The treatments primarily removed hemicelluloses along with some lignin. This was shown to impact a variety of physical and chemical properties of the wood including intra cellwall porosity, cellulose crystallinity, wood surface energy, equilibrium moisture content, and heat content. These fundamental changes manifested in a variety of changes to properties of OSB manufactured from the extracted material. OSB exhibited improved dimensional stability water vapor, no change in thickness swelling due to water immersion, and an increase in water absorption. The flexural strength and modulus was not significantly changed for the intermediate weight loss condition, but was decreased at the more severe weight loss condition. In all cases, internal bond strength was reduced. Decay resistance to white rot fungi was improved while no impact was observed in brown rot fungi resistance. Future studies should look at weight loss conditions less than 15% to minimize wood cell wall damage.

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