MODIFICATION OF WOOD FIBER USING STEAM

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ABSTRACT

High temperature steam treatment of wood fiber in a closed press during fiberboard pressing and then cooling the fiberboard while still under pressure to below the glass transition temperature of lignin, greatly increased the dimensional stability and decreased the hemicellulose content of the fiberboards produced. For example, after pressing aspen fiber four minutes at 200 C under steam pressure, the resulting fiberboards show less than 10 percent thickness swelling after two-hour water soak as compared to over 40 percent for non-steamed fiberboards. The addition of ferric chloride to the process greatly increases dimensional stability and reduces the hemicellulose content lower than control fiberboards.

INTRODUCTION

High temperature steam treatment of wood results in several changes in the chemistry of wood. These include: (1) degradation of the hemicelluloses to produce simple sugars which may undergo reversion reactions to form highly branched polysaccharides, (2) degradation of both the hemicelluloses and part of the cellulose to form furan type compounds, (3) thermal softening of the cell wall matrix, mainly lignin, (4) degradation of the hemicelluloses to form volatile break down products, (5) crosslinking between carbohydrate polymers and/or between lignin and carbohydrate polymers, and/or (6) an increase in cellulose crystallinity. While no definitive mechanism has been published, there are clues in the literature to support one or more of the above theories.

The research reported in this paper was done to treat wood fiber, in a closed press, with high temperature steam to (1) degrade the hemicelluloses in order to decrease hygroscopicity and equilibrium moisture content, (2) generate furan monomers that could form polymers for increased internal bond strength, (3) thermoplastaicize the lignin matrix and reform it in a cool down process to generate a new collapsed unstressed matrix, and (4) produce a fiberboard with greatly increased dimensional stability.

BACKGROUND

Treating wood with heat dates back into the oral history of Africa. Wooden sticks destined to become spears were heat treated to harden and sharpen them. The treatment involved several steps including placing the end of the stick under hot coals, withdrawing after a certain time and then hitting the burned stick end with a rock. This process was repeated many times until the end was hard and sharp. The mechanism of this process is quite simple. Heating wood in the absence of oxygen caused the wood to undergo pyrolysis converting some of the hemicellulose and cellulose sugars to furan resins and some of the wood substance to charcoal. Pounding the pyrolyzed stick end with a rock drove the furans and charcoal into the interface/interphase of the charred under-wood surface. Repeating this process and shaping the end resulted in a furan impregnated, carbon hardened point.

Early Native Americans in the Pacific Northwest made fish hooks of wood using steam. Small green wooden sticks were cut to length and sharpened. The sticks were placed in a kelp tube and the tube filled with water. The tubes were pluged with moss and buried in hot ashes of a dying fire all night. The next morning the kelp was split open and the steamed wood was bent into the shape of a fish hook. The wet and hot wood was pressed in a wooden mool and left to cool. The bent hooks were then heated again dry and rubbed with deer tallow and put back into the mold to cool again. Bone barbs were lashed onto the hook using spruce or cedar roots.

In the 1930's, Stamm and coworkers started a long series of studies on heat stabilization of wood (Stamm and Hansen 1937). Stamm's work was based on earlier research of Tiemann (1920) who showed that high-temperature kiln drying of wood decreased the hygroscopicity and subsequent swelling and shrinking of lumber. Heating wood in a vacuum at high temperatures caused lignin to flow and the hemicelluloses to decompose producing water-insoluble polymers. This treatment increased stability but decreased strength. One such treatment was known as Staybwood (Stamm et al. 1946). Staybwood was made by heating wood at temperatures between 100-160 C° in a bath of molten metal (50% tin, 30% lead and 20% cadmium) with a melting point of about 150 C°. This alloy did not stick to the wood surface. Sand circulated with dry nitrogen was also used to heat veneers with similar results. Heating times ranged from a few minutes at high temperatures to a few hours at lower temperatures. Dimensional stability of Staybwood increased as both heating time and treating temperatures were increased, whereas strength decreased. Under conditions that reduced swelling and shrinkage by 40%, toughness was reduced to the same extent. Abrasion resistance was also reduced. The hygroscopicity of Staybwood was greatly reduced and resistance to decay was improved.

The use of high levels of heat alone to improve dimensional stability of wood can cause extensive damage to the cellular structure. For this reason, researchers also looked at heat stabilization using both heat and moisture. Seborg et al. compressed wood while heating moist wood to form a heat stabilized product known as Staypak (Seborg et al. 1945). Staypak was made by compressing thin veneers that had been conditioned to 30 to 65% relative humidity at temperatures between 170-177 C°. The compressed veneers had a specific gravity of about 1.3. The compressed product sorbs water at a slower rate than uncompressed wood and was more dimensionally stable. It was not more resistant to biological attack. Strength properties were much higher than untreated wood. Strength increased almost proportionally to the amount of compression. Tensile strength both parallel and perpendicular to the grain, modulus of rupture, and elasticity in bending were increased. The greatest increase was in impact bending strength. Other products similar to Staypak were produced in Germany called Lignostone and Lignofol and in England Jicwood and Jablo (Rowell and Konkol 1987).

Burmester (1973) also observed that swelling could be reduced in wood based on heating wood in a sealed system under pressure. He compressed oak, beech, spruce and pine with moisture contents ranging from 20 to 30% and found 52-75% reductions in deformation due to moisture regain. Similar results were found for particleboard.

Giebeler (1983) treated wood at 180 to 200 C° in the presence of moisture and found that these treatments resulted in a large reduction in the resistance to shock, modulus of elasticity (MOE), modulus of rupture (MOR) and compression strength. Hillis (1984) also studied the effects of high temperature and chemicals on wood stability. Hsu (1986, 1988) published data on a new process using high pressure steam digestion of aspen and pine flakes and fiber before board production. Flakes or fiber was hot pressed at 177 C° for 9 minutes followed by 4 hours at 138 C° in a forced air oven. During the process, he found that the xylan content was reduced, free xylose was formed, water solubles increased, and both lignin and cellulose content remained unchanged using aspen flakes. Skaar (1976) reported that heating wet wood degraded 10 times faster than heating dry wood.

Viitaniemi and Jamsa (1994) also studied heat treatments of wood to improve dimensional stability. Inoue et al. (1992, 1993) used a high pressure, high temperature treatment to stabilize solid wood and Rowell et al. (1998) used a similar process to stabilize fiberboards made using jute fiber. In both of the research of Inoue and Rowell, the steam used in the process is generated from moisture in the wood or fiber during the hot pressing and contained in the hot press by means of a rubber gasket which contains the wood or fiber during the process. Also, in both of these approaches the wood or fiberboard produced is cooled in the press under pressure (Rowell et al. 1998).

In 1997, Honda patented a process where wood, below the fiber saturation point is heated, under pressure, to a temperature above the softening temperature (Ts) of cellulose with decompression followed by recompression. A similar process of compression, decompression and recompression at high temperatures applied to dry wood was patented by Hasegawa and Tomita (1997).

Another process has been reported called the PLATO process (Proving Lasting Advanced Timber Option) Tieerdsma et al. 1998). In this three step process, green wood is heated under pressure between 160 to 190 C°, it is then dried to below 10% moisture by conventional drying and then cured between 170 and 190 C° at atmospheric pressure in the absence of air. The final product has a lower hygroscopicity and improved dimensional stability as compared to controls.

Jones (1999) has reviewed the literature on treatments of wood and plant fibers using chemicals, pressurized thermal and steam treatments.

Wise and John (1952) report a wide range of thermal degradation products from wood. It is known that the hemicelluloses are much less stable to heat and undergo thermal degradation more rapidly than cellulose. (Stamm 1964). Mitchell et al. (1953) reported that the hemicelluloses in Douglas fir decomposed at 220 C° faster than alpha-cellulose which was faster than lignin. Interestingly, decomposition was faster and occurred to a greater extent in a closed system as compared to an open system. They also found that, under these conditions, the hemicelluloses decomposed mainly to carbon dioxide and water with the formation of very little condensation products. Melcer et al. (1990), however, found that the hemicelluloses degraded the fastest but that lignin was degraded faster than cellulose in beech wood at 120 C°. Sondermann and Augustin (1964) reported pentosan degradation varied from 8% at 170 C° to 100% at 350 C°. A review of the thermal degradation of wood has been published by Stamm (1964), Shafizadeh (1984), and Fengel and Wegner (1984).

Fengel and Wegner (1984) reported that organic acids were formed as reaction products from the non-hydrolytic degradation of hemicelluloses. Kosik et a1 (1968) reported that acetaldehyde, methyl acetate, propyl aldehyde, acetone, acetic acid, methanol and furfural were formed from the thermal decomposition of 4-0-methylgluconoxylan. Aoyama (1996) reported that a large percentage of the hemicelluloses from bamboo were solubilized at temperatures between 170 and 206 C°. The extent of degradation was dependent on moisture content, temperature and time of treatment. Schmidt et al. (1996) reported that there were differences in the degradation of the hemicelluloses in the steam treatment of wheat straw as compared to birch wood. It was found that the optimum conditions for degrading hemicelluloses, by steam, from straw were significantly different than those used for wood.

Several researchers have shown that the yield of free sugars formed from hemicelluloses during steaming of lignocellulosics increase undergo a maximum in concentration and then decrease with increasing time of steaming (Oji and Mowat 1978, Rangnekar et al. 1982, Overend and Chornet 1987, Lawther et al. 1996). This would indicate that free sugars released from the degradation of the hemicellulose then undergo further reactions to other products. Pectic polysaccharides have also been shown to influence the production of free sugars upon heating (Overend and Chornet 1987, Lawther et al. 1996).

Past research done by the Masonite Corporation also provides useful information on the chemical changes when wood is heated with steam (Mason 1926, Boehm 1940). Fiberization was done at high temperatures and pressurized steam. The yield of fiber from this process was approximately 80 to 85% (Turner 2000). The fiber then went through three counter current washing cycles to remove soluble sugars. The additional fiber weight loss after washing was 10 to 15%. If the concentration of sugars in the fibers before pressing was too high, the fiberboards would stick in the press. If the sugar content was too low, the fiberboards would not stick together. During the counter current wash cycles, furfural could be detected by smell, however, no analytical determination of the exact amount generated was ever carried out. The weight loss during refining was assumed to be mainly due to degradation of the hemicellulose polymers producing both free sugars and other degradation products, including furfural. During the hot pressing in the production of fiberboards, the free sugars underwent reversion reactions under the high temperature acidic conditions giving rise to complex, highly branched polysaccharides. The hygroscopicity of the reversion derived polysaccharides was lower than the other cell wall polymers resulting in a reduced hygroscopicity of the final fiberboard. The counter current washing removed most of the soluble sugars. Inoue and Norimoto also reported the formation of furfural during the heating of moist wood (Inoue et al. 1991, Norimoto et al. 1994).

Shen describes another process for degrading part of the hemicelluloses using a steam explosion process of wood chips to produce fiber (Shen 1985). He claims, but does not prove, that furan monomers are formed in the process. Using this process, Brooks has shown that heating wood at 208 C° for 1 to 3 minutes and exploding in a Stake Digester results in the release of most of the pentasans in the hemicelluloses (Brooks 2000). Using only the fibers from the digester, boards were made which showed less that 9% thickness swelling after a 2 hour boiling test. Approximately half of the fiberboard strength properties remain after this boiling test.

It is well known that lignin and the hemicelluloses are amorphous polymers. Cellulose, on the other hand, is mainly crystalline but does contain regions with amorphous character within the crystalline lattice. Amorphous polymers undergo changes in physical characteristics upon heating resulting in transitions to a glassy and rubbery state (Irvine 1985). The temperature where this happens is referred to as the glass transition temperature or Tg. Transitions from one state to another depend on sufficient energy input to overcome the intermolecular attractive forces with in the polymer and allow a greater degree of flow. Chawla and Sharma showed that once the temperature exceeded 160 C°, there was sufficient flow of lignin to block pores within the wood structure to restrict the reentry of moisture (1972). Hillis and Roza (1978) found that a minimum heating of 2 hours at 100 C° plasticized the hemicellulose-lignin matrix.

It is also well recognized that moisture lowers the Tg of an amorphous polymer (Goring 1963). Goring examined the softening temperatures for dry and wet lignin, hemicellulose and cellulose and found that the sorption of water greatly decreased the Tg of both lignin and hemicellulose while the Tg for cellulose only varied slightly from dry to wet. The Tg for dry lignin is approximately 155-160 C° depending on the chemical structure. High syringyl content lignins have a lower Tg as compared to other lignins (Olsson and Salmen 1993). Inoue and coworkers showed that at temperatures between 180 and 200 C°, the lignin-hemicellulose matrix became plasticized and underwent thermoplastic flow (Inoue et al. 1993). All of this data shows that under heat and pressure, the lignin-hemicellulose matrix becomes thermoplastic and will deform to a different stable matrix. If the compressed matrix is cooled under pressure, the newly formed matrix has little memory of its precompressed form (Rowel1 et al. 1998).

Chawla and Sharma published results suggesting that during the heating process crosslinking of polysaccharide chains could occur (1972). They also suggested that some of the thermal degradation products recombined during heating. Norimoto (1994) and Dwianto et al. (1998) also suggested the formation of interlinkages between wood polymers during the heat treatment of wood. Boonstra et al. (1998) suggested that degradation products from the hemicelluloses and lignin for reactive intermediates that cross link during the curing stage in the PLATO process (see above).

Finally, it has been shown that heating wood under pressure resulted in an increase in cellulose crystallinity (Inoue et al. 1991). This could also have an influence on rewetting of the cell wall polymers after compression.

Based on the literature review, there are many possible chemical reactions taking place during steaming of wood fiber. Of interest to our research program is the loss of hemicelluloses (to decrease hygroscopicity and increase resistance to attack by micro-organisms) and to convert the released free sugars to furan intermediates (that can be converted to furan resins to increase internal bond strength) and to increase in dimensional stability of fiberboards. The effects of the treatments depend on many variables such as temperature, time of treatment, moisture content, chemical composition of the lignocellulosic, the addition of specific catalysts, and probably other factors which are not yet understood.

EXPERIMENTAL

The pressing system used in this research was described previously (Rowell et al. 1998, 2000). A flat silicone ring 5 cm on each side with a 23 cm center cut out. The rubber ring was placed in a 35 by 35 cm press heated by oil and cooled by water.

Steam Treated Fiberboards

Aspen fiber was dried over night at 105 C° and weighed. A water spray was used to bring the fiber moisture content to 15%. A hand formed fiber mat was formed inside the pressing ring and then placed in the press. The press was then closed to come into contact with the rubber ring to contain the steam generated from the moisture in the fiber. The fibers were pressed at 200 C° for 4,8 or 16 minutes to form fiberboards with a specific gravity of about 1.0. A known amount of fiber was used for each board and the press was closed to metal stops to give the desired specific gravities. After pressing, the fiberboards were cooled, under pressure, in the press, removed, oven dried, weighed and cut into test specimens.

Modified Steam Treated Fiberboards

Similar fiberboards were made as given above except the fiber was sprayed with either ferric chloride [0.5% (pH 1.2), 1% (pH 1.4, or 2% (pH 1.6) solutions] to bring the fiber moisture content to 15%. These boards were pressed for 4,8 or 16 minutes at 200 C° to give fiberboards with a specific gravity of about 1.0. After cooling in the press, under pressure, the fiberboards were removed, oven dried, weighed, and cut into test specimens.

Water-Soaking and Thickness Swelling Tests

Oven dried, weighed and thickness measured specimens (5 by 5 cm) were placed in a container of distilled water, soaked for 24 hours at room temperature and the increase in thickness determined. Five specimens of each board were tested and the results averaged.

Sugar Analyses

Sugar analysis was done as given before (Rowel1 et al. 2000).

RESULTS AND DISCUSSION

Because of the limited number of specimens per individual test, statistical analysis of the date was not appropriate. The results presented here should be considered as indicative of trends that a larger, statistically valid experiment should confirm.

Thickness Swelling

Table 1 shows the increase in fiberboard thickness as a result of water soaking. Thickness swelling decreased in control fiberboards as press time increased. Thickness swelling decreased in ferric chloride treated fiberboards both as a function of solution concentration and pressing time. Treatment with a 2% solution of ferric chloride for 4, 8, 16 minutes pressing time resulted in thickness swelling of 7, 5, and 4.3% respectively. A pressing time of 4 to 8 minutes is compatible with the present fiberboard pressing cycle.

Table 1. Thickness Swelling of Steam Treated Aspen Fiberboards

Specimen	**Increase in Thickness Swelling (%)**		
	4 min	8 min	16 min
Control	17.7	14.0	11.3
Ferric Chloride Treated			
0.5%	14.0	7.9	3.9
1%	10.2	5.7	4.1
2%	7.0	5.0	4.3

Sugar Analysis

Table 2 shows the sugar analysis before and after steam treatment. Comparing the sugar analysis before and after steam treatment, it can been seen that steaming for 4 minutes results in a major loss of arabinan with minor losses of rhamnan, glucan and mannan. Steaming for 16 minutes, results in an almost complete loss of arabinan and rhamnan with major losses of galactan and xylan. Glucan and mannan fractions are very stable to steaming. Adding ferric chloride to the system and steaming for 4 minutes results in even more loss of arabinan, galactan, rhamnan, xylan, and mannan as compared to control steamed at 4 minutes. After 16 minutes steaming in the presence of ferric chloirde, all of the arabinan, galactan, rhamnan are removed and most of the xylan and mannan are removed. The glucan is stable to steaming in the presence of ferric chloride.

Table 2. Sugar Analysis Before and After Steam Treatment

Specimen	Arabinan	Galactan	Rhamnan	Glucan	Xylan	Mannan	Total
Control							
Before Steam Treatment							
	0.53	0.60	0.32	43.50	16.38	1.65	63.0
After Steam Treatment							
4 minute	0.28	0.59	0.22	42.10	15.98	1.56	60.7
8 minute	0.20	0.58	0.14	42.16	14.63	1.49	59.2
16 minute	0.04	0.24	nd	45.21	9.08	1.34	55.9
Ferric Chloride (2%) Treated After Steam Treatment							
4 minute	0.11	0.33	0.13	43.18	9.47	1.04	54.3
8 minute	0.03	0.13	nd	43.76	2.90	0.50	47.3
16 minute	nd	nd	nd	43.01	0.87	0.16	44.0

CONCLUSIONS

In earlier research (Rowel1 et al. 2000), we found that both the rate and extent of thickness swelling could be reduced and the equilibrium moisture content was reduced using the closed steaming process. It was also shown that hemicellulose sugars were lost during the steaming and furan intermediates were formed. There was no attempt to detect or determine furan monomers in this study, however, this is presently under investigation. The addition of ferric chloride to the system greatly increased the degradation of hemicellulose sugars and reduced the thickness swelling even more that the system without using ferric chloride. Present studies in this area are directed at both the mass balance and the rate of hemicellulose sugar loss and the rate of furan intermediate formation. Other chemical additives, which may be more effective than ferric chloride, are also under study. Mechanical properties of the fiberboards are also being studied to determine strength losses, if any, which may occur as a result of using a chemical such as ferric chloride in the system.

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