Latest Advancements in The Acetylation of Wood Fibers To Improve Performance of Wood Composites

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ABSTRACT

A new procedure has been developed for the rapid continuous acetylation of lignocellulosic fiber. A limited amount of acetic anhydride is applied to the fiber before the fiber goes through a reactor at high temperature. The acetylated fiber is then stripped in a first step with superheated vapor of anhydride/acetic acid and, optionally, in a second step with superheated steam to both dry the fiber and convert any un-reacted anhydride to acid making separation of byproducts easier. Fiberboards made using the acetylated fiber were much more stable to dimensional changes in both high relative humidity and liquid water without loss of strength properties as compared to non-acetylated fiberboards.

Keywords: Fiber, acetylation, dimensional stability, equilibrium moisture content, reaction conditions, strength properties.

INTRODUCTION

While our attention is being drawn to renewable, biodegradable and sustainable resources for materials, we are faced with the concern about durability of products used in adverse environments. For the most part, we have designed and used low cost wood fiber-based composites accepting limitations such as dimensional instability, ultraviolet and biological degradation, and thermal instability. However, with a better understanding of the relationship between chemistry, properties, and performance of wood fibers, we can produce a new generation of value added composites that will be performance driven and durable.

Properties such as dimensional instability, flammability, biodegradability, and degradation caused by acids, bases, and ultraviolet radiation are all a result of chemical degradation reactions (hydrolysis, oxidation, dehydration, and reduction) which can be prevented or, at least, slowed down if the cell wall chemistry is altered. This approach is based on the premise that the properties of any resource are a result of the chemistry of the components of that resource. In the case of wood fibers, cell wall polymers, extractives, and inorganics are the components that, if modified, would change the properties of the resource. Based on performance requirements, chemical modifications can be carried out to change the chemistry of the resource that will result in a change in performance.

Many chemicals have been used to chemically modify wood fibers to improve performance including anhydrides, acid chlorides, carboxylic acids, isocyanates, aldehydes, lactones,

nitriles, and epoxides (Rowell 1975, 1983, In Press, Rowell and Ellis 1981, 1984, Rowell et al. 1986). Attention in recent years, however, has focused on acetylation chemistry. This paper presents a new procedure for the acetylation of wood fiber and properties of composites made using this fiber.

ACETYLATION TECHNOLOGY

The new process can be broken down into four basic steps: addition of chemical, reaction, and stripping of surplus acetic anhydride and formed acetic acid in two steps (Nelson et al. 1994. 1995a,b). A measured amount of preheated acetic anhydride is mixed with the dried wood fiber in a continuous moving screw reactor at approximately 140 °C with a residence time of 5 to 10 minutes. A plug screw at the end of the reactor separates the reactor from the first stripping tower. The atmosphere moving the fibers through the first stripper consists of a mixture of anhydride and acetic acid vapors. The vapors are kept superheated by a steam-heated exchanger and the fibers are separated in a cyclone before being discharged to the next stripping step. Superheated steam (water vapor) is used in the second stripping tower to convert un-reacted anhydride to acid. The dry fiber is then removed form the system and ready for adhesive application if the acetylated fiber is to be made into composites.

TEST PROCEDURES

Fiberboard fabrication using phenolic resin

Control or acetylated pine fibers were sprayed in a laboratory blender with a water soluble, 45% solid content, liquid phenolic resin. The fiber was then run through a hammermill to fluff and eliminate clumps. A final resin content of 8% was used (based on moisture free weight). The blended fibers were air dried to final moisture content of 5-7%. The fibers were then hand formed into a randomly oriented mat and pressed for 8 minutes at a press temperature of 180 °C to form fiberboards with specific gravities of approximately 0.9-1.0.

Equilibrium moisture content (EMC) and thickness swelling (TS)

Weighed and oven-dried control and acetylated fiberboard specimens (5 by 5 cm) were placed in constant humidity rooms at 30%. 65% and 90% relative humidity at 27 °C. The specimens were left until reaching a constant weight, then weighed to determine the EMC and the thickness measured to determine thickness swelling (TS). Equilibrium moisture content (EMC) was determined based on oven dry specimen weight. Five specimens of each set of fiberboards were tested and the results averaged.

Water-soaking and thickness swelling tests

Oven-dried, weighed and thickness measured control and acetylated fiberboard specimens (5 by 5 cm) were placed in a container, distilled water was added and the board thickness was recorded as a function of time. Wet measurements were taken every 15-minute for the first hour, every hour for the first 5 hours, then once a day for 5 days. In a separate test, specimens were removed after 24 hours, re-ovendried for 24 hours at 105 °C, and thickness determined. Five specimens of each set of recycled boards were tested and the results averaged.

Strength tests

Static bending tests were conducted on board specimens (5 by 20 cm) conditioned to 65% relative humidity according to ASTM standard D 1037. Modulus of rupture (MOR) and modulus of elasticity (MOE) were determined. Ten specimens from each board were tested and the results averaged.

RESULTS AND DISCUSSION

Table 1 shows the results of the equilibrium moisture content and thickness swelling tests. The EMC for control fiberboards ranged from 5.8% at 30% RH to 21.7% at 90% RH and thickness swell ranged form 1.0% at 30% RH to 11.2% at 90% RH. The fiberboards made using acetylated fiber had EMC values of 2.4% at 30% RH and 8.4% at 90% RH with thickness swelling of 0.2 at 30% RH and 2.9% at 90% RH. This data shows that the moisture content of acetylated fiber is much lower than control fiber at a given relative humidity.

Table 1 - Equilibrium moisture content (EMC) and thickness swell (TS) of control and acetylated pine fiberboards (27 °C, in %)

WPG	30% RH		65% RH		90% RH	
	EMC	TS	EMC	TS	EMC	TS
0	5.8	1.0	12.0	3.1	21.7	11.2
20.7	2.4	0.2	4.3	1.4	8.4	2.9

Table 2 shows the data from the water soaking rate test. Control fiberboards start to swell immediately upon contact with water and after just 15 minutes of water soaking have swollen 25.7%. This swelling continues to occur and after 5 days, the swelling has increased to 36.2%. Acetylated fiberboards swell very slowly and even after 5 days, the swelling is less than 5%.

Table 2 - Rate and extent of thickness swelling in liquid water of fiberboards made from control and acetylated pine fiber (in %).

WPG	Percent Thickness Swelling at						
	Minutes			Hours			Days
	15	30	60	3	6	24	5
0	25.1	29.8	33.5	33.8	34.0	34.0	36.2
21.6	0.6	0.9	1.2	1.9	2.5	3.7	4.5

Table 3 shows the residual swelling in the fiberboards after 24 hours of water soaking and re-ovendrymg. The control pine fiberboard showed 34% thickness swelling when wet for 24 hours and after re-drymg was still 22.7% thicker than the original dry fiberboard. During the same test, fiberboards made from acetylated fiber on swelled 3.7% in 24 hours and were only 1.9% thicker after drying as compared to the original dry fiberboards.

Table 3 - Thickness swelling of control and acetylated pine fib3er boarads in water and thickness after drying (ln %)

WPG	Thick swelling	Residual Thickness Swelling
	(24 hours in water)	(oven dry)
0	34.0	22.7
21.6	3.7	1.9

Table 4 shows the results of the strength test on control and acetylated fiberboards. There is not statistical difference between the two sets of fiberboards.

Table 4 - Modulus of rupture (MOR), modulus of elasticity (MOE), and internal bond strength (IBS) of fiberboards made from control and acetylated pine fiber (8% phenolic resin)

WPG	MOR	MOE	IBS
	(MPa)	(GPa)	(MPa)
0	53	3.7	2.3
19.6	61	4.1	2.3

CONCLUSIONS

Acetylation using the new fast continuous acetylation procedure produces acetylated fiber that can be processed into fiberboards with greatly reduced equilibrium moisture content, increased dimensional stability with less residual swelling after wetting as compared to control fiberboards. The strength properties are not reduced as a result of acetylation.

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PROCEEDINGS

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Statement of Procedure