DECAY PROTECTION BASED ON MOISTURE EXCLUSION RESULTING FROM CHEMICAL MODIFICATION OF WOOD

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ABSTRACT

Southern pine solid wood (SPS) and fiber (SPF) were chemically modified to various weight percent gains (WPG) using either acetic anhydride (AA, 4%-19% WPG), butylene oxide (BO, 4%-23% WPG), or propylene oxide (PO, 6%-30% WPG). After modification, part of the specimens were extracted with a toluene:ethanol (2:1) solution for 2 hours or water leached for 2 weeks. The equilibrium moisture content (EMC) at 30%, 65% and 90% relative humidity (RH) and 27 ℃ was determined on all specimens. Laboratory soil block decay testing using the brown-rot fungus *Gloeophyllum trabeum* was performed and weight loss calculated.

Two possible mechanisms for biological efficacy by chemical modification of wood have been proposed. One involves lowering the cell wall moisture content below a level required for microorganism attack. The other involves modification of the substrate in such a way that the specific enzymatic reactions cannot take place. Results on solid wood indicate that as the EMC is lowered by chemical modifications, biological protection increases. Chemically modified fiber shows a similar trend with AA and BO modifications, but not with PO. Modification of wood fiber with PO does not lower the EMC significantly, even at the highest WPG, yet it imparts biological resistance. This indicates that the mechanism of efficacy may be due to substrate modification.

Keywords: Chemical modification, acetylation, butylene oxide, propylene oxide, equilibrium moisture content, decay

INTRODUCTION

Untreated wood with low natural durability that is exposed to outdoor applications often biologically decays. Therefore, wood preservatives are applied to protect wood products to extend their lifetime. There are numerous ways to protect wood from biological degradation. Today's most common method today is by applying toxic chemicals. This is very effective, but with environmental concerns, alternatives are being considered. Chemical modification of wood has potential as an alternative for protecting wood from biological degradation. The modifications are not based on introducing biocides or toxicity and therefore should be more environmentally friendly. It can increase dimensional stability and biological resistance (Rowell 1983).

Wood is a three dimensional polymeric composite made up of lignin, hemicelluloses, and cellulose. The cell wall is made up of these three polymers, which determine the physical and chemical properties of wood. Each of these three polymers has accessible hydroxyls and other oxygen containing groups that attract moisture through hydrogen bonding making wood hygroscopic (Rowell and Banks 1985), (Stamm 1964). Wood then will sorb and desorb moisture to maintain an equilibrium with its environment. Each wood component sorbs moisture to a different extent: hemicelluloses > cellulose > lignin (Christensen and Kelsey 1959), (Rowell 1982), (Rowell and Rowell 1988).

Chemically modifying the cell wall polymers changes the hydrophilic nature of wood. Chemical reagents react with the cell wall hydroxyl groups resulting in stable, covalent bonds. Various chemical modifications and procedures for wood modification have been reviewed (Rowell 1983), (Kumar 1994), (Militz et al. 1997).

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Two possible mechanisms for biological protection by chemically modifying wood have been proposed. One involves lowering the cell wall moisture content and bulking the cell wall with chemicals that react with hydroxyl groups. Chemical modification blocks many of the accessible hydroxyls of the wood cell wall through chemical bonding therefore eliminating the moisture sorption sites. The decay fungi responsible for wood deterioration are dependent upon moisture for their existence. Literature states that serious decay occurs when the moisture content of wood is above the fiber saturation point (average 30%). Thus, chemical modification lowers the cell wall moisture content below the level required for microorganism attack.

The other mechanism involves a change in the configuration and conformation of the food source or substrate modification. The altered chemical configuration does not allow the enzymes to fit the substrate in a "lock and key" type arrangement (Zubay 1983). The substrate is modified in such a way that the specific enzymatic reactions cannot take place. Wood is degraded biologically because fungi recognize the carbohydrate polymers (mainly hemicelluloses) in the cell wall. Decay fungi have very specific enzyme systems capable of hydrolysing the polymers into digestible units. Therefore, the key to brown-rot fungal resistance may lie in the protection of the hemicellulose polymers; if they are protected by chemical modification, attack cannot proceed.

Chemically modified composites have been tested with decay fungi in several ways. Untreated controls and chemically modified particleboards were exposed to a 12 week soil block test using the brown rot fungus *Gloeophyllum trabeum* and the white rot fungus *Trametes versicolor*. All boards were made using a phenolic resin (Nilsson et al. 1988), (Rowell et al. 1988a). All of the bonded chemicals at a WPG over about 20 show good resistance to brown- and white-rot fungi except propylene oxide in the brown-rot test. Propylene oxide was not effective in preventing attack by brown-rot fungi even though the same number of hydroxyl groups should be modified as were modified by reaction with butylene oxide, methyl isocyanate, acetic anhydride, b propiolactone or acrylonitrile (Rowell et al. 1988b).

Southern pine solid wood was chemically modified with acetic anhydride, propylene oxide, butylene oxide, methyl isocyanate and n-butyl isocyanate (Ibach and Rowell 1999). Samples were analyzed for equilibrium moisture content (at 30%, 65%, or 90% and 27 °C), fiber saturation point (non-solvent water technique (Feist and Tarkow 1967)),and biologically with both the soil block test (ASTM 1976) and fungal cellar (Nilsson and Rowell 1982). Results indicate that acetylated wood lowered the EMC, FSP and also showed biological protection at 14.8% weight gain in the soil block test and 19.1% weight gain after 12 months in the fungal cellar. Methyl isocyanate, n-butyl isocyanate, and butylene oxide lowered the EMC and were biologically effective, but at higher weight percentage gains. Propylene oxide was not effective in lowering the EMC nor in protecting the wood specimens biologically against *G. trabeum.* This exception of propylene oxide to the protection rule is perhaps the key to understanding the mechanism of the resistance to attack by fungi by chemical modification. The EMC of propylene oxide modified wood was higher than the other modified wood and this may be the reason for the lower biological resistance.

in this paper the correlation between EMC and biological resistance of southern pine solid wood and fiber chemically modified with acetic anhydride (AA), butylene oxide (BO), and propylene oxide (PO) was examined. The overall aim of the research was to determine if lowering the EMC to a certain point by chemical modification would lead to fungal resistance.

MATERIALS AND METHODS

Chemical Modification

Southern pine softwood solid wood (2.54 cm x 2.54 cm x 0.64 cm) and southern pine fiber were chemically modified with three different chemicals: 1.) acetic anhydride, 2.) butylene oxide or 3.) propylene oxide. Acetic anhydride and butylene oxide were from Eastman Chemical Company (Kingsport, TN) and propylene oxide and triethylamine (the catalyst) were from Aldrich Chemical Company (Milwaukee, WI).

Acetic Anhydride:

Solid Wood:

Southern pine specimens (2.54 cm x 2.54 cm x 0.64 cm) were cut and dried at 105 °C in a forced draft oven for 24 hours. Samples were weighed and then reacted in a glass reactor from 3 minutes up to 240 minutes with acetic anhydride (Goldstein et al. 1961). Reaction temperature was 120 °C. After modification, samples were again oven dried and weighed. Weight percent gain was determined from original oven dried weights. Selected samples were water leached for 14 days (AWPA 1999).

Fiber:
Southern pine fiber was screened, washed, and then oven dried before treatment. The fiber was dipped in acetic anhydride for 1 minute. Excessive solution was drained for 5 minutes. Fiber was placed in a one-liter glass reactor that was heated with an oil bath at 120-125 °C from 22 minutes up to 240 minutes. The treated fiber was oven dried and weight gain percentage (WPG) calculated. Selected samples were water leached for 14 days (AWPA 1999).

Acetyl Analysis:
Acetyl content was determined on acetylated solid wood and fiber specimens, as well as untreated controls using the following method (Davis 2000). Specimens were milled to pass a 20-mesh screen, vacuum dried at 45° C, and treated under conditions to maximize liberation of acetyl groups and minimize production of acetate via sugar degradation. Liberated acetate was measured by anion exchange high performance liquid chromatography using suppressed conductivity detection. Propionic acid was used as an internal standard. The chromatographic system consisted of a 738-auto sampler (Alcott Chromatography, Norcross, GA), a GP40 quaternary gradient high-pressure pump (Dionex Corporation, Sunnyvale, CA), and a CDM-1 conductivity detector (Dionex). Ion Pac AG-11 guard and AS-11 analytical columns were connected in series and eluted with 0.10 mM NaOH at a flow rate of 2.0 ml/min. Prior to each injection, the columns were conditioned with 40 mM NaOH and equilibrated with 0.10 mM NaOH. Post-column neutralization of the eluent stream prior to conductivity detection was accomplished with an ASRS-Ultra 4mm membrane suppressor and coupled SRS-Controller (Dionex) operating in auto suppression recycle mode. Under these conditions, acetate and propionate eluted at *ca.* 4.7 and 5.5 min, respectively.

Propylene Oxide (PO) and Burylene Oxide (BO):

Oven dried solid wood southern pine specimens (2.54 cm x 2.54 cm x 0.64 cm) and southern pine fiber were weighed and then reacted in a stainless steel reactor with a mixture of propylene oxide or butylene oxide and triethylamine (95:5 (vol.:vol.)) at 11 0 or 120 °C and 150 psi nitrogen pressure, from 5 to 75 minutes for propylene oxide and 20 minutes to 6 hours for butylene oxide (Rowell and Gutzmer 1975). The treating solution was drained off. Samples were air dried under a fume hood and then oven dried. Selected samples were either water leached for 14 days (AWPA 1999) or extracted in a soxhlet extractor for 2 hours with toluene:ethanol mixture (2:1, vol.:vol.) Percent weight gain was calculated for unleached, water leached, and solvent extracted samples.

Equilibrium Moisture Content (EMC)

EMC of untreated and chemically modified wood samples was determined by placing weighed, oven dried samples in constant humidity rooms at 30%, 65%, or 90% relative humidity (RH) and 27 °C. After 10-14 days samples were reweighed until stable and the EMC was determined. Six replicates of each treatment were run and averaged.

Biological Efficacy

The ASTM D 1413 standard soil block test was performed on both solid wood and fiber specimens (ASTM 1976). Untreated controls and chemically modified specimens of southern pine solid wood and fiber (0.5 g) were exposed to the brown-rot fungus *Gloeophyllum trabeum.* Fiber was removed after 9 weeks and the solid wood after 12 weeks. The extent of decay was determined as oven dry weight loss.

RESULTS AND DISCUSSION

Chemical Modification

Table 1 shows the reaction times as well as the weight percentage gain (WPG) and percentage acetyl content of unleached and water leached acetylated specimens. The data suggests that there is negligible weight loss and acetyl loss after 2 weeks of water leaching with both the solid wood and fiber. The untreated solid wood and fiber that were water leached showed a weight loss of - 1.6% and –3.9%,respectively. This may be explained by loss of the hemicellulose component that can be removed in the acetylation procedure.

Table 2 and Table 3 show the reaction times as well as the WPG of the unleached, toluene:ethanol extracted and water leached of the BO and PO specimens. The PO modified solid wood specimens have significant weight loss (about 6%) with water leaching and less with the toluene:ethanol extraction. The BO modified solid wood specimens have about half the weight loss (about 3%) as that of PO with water leaching and toluene:ethanol extraction. The BO and PO modified fiber has almost negligible weight loss. This difference between solid wood and fiber may due to the fact that the fiber was water leached before treatment or that the accessibility of the reactive hydroxyl sites is greater with the fiber than the solid wood.

The reaction temperature has an affect on the leaching amount with BO and PO modified solid wood, but not with the fiber. At 120 °C there is less weight loss after water leaching and solvent extraction than at 110 °C after 6 hours

with the solid wood. The fiber is not affected. This indicates that there is better accessibility of the hydroxyl sites with fiber than solid wood. It also indicates that the epoxides polymerized in the cell lumen at the lower temperature during the reaction with solid wood (Rowell and Ellis 1984).

Equilibrium Moisture Content and Biological Efficacy

The EMC at 30%, 65%, and 90% RH, and 27 °C of southern pine solid wood and fiber modified with AA, BO, and PO is presented in Table 4. The fiber for all three modifications has a lower EMC than the solid wood samples. The water leached solid wood samples have a higher EMC than the unleached, but it seems insignificant in the acetylated fiber samples. This may be explained by the loss of the hemicelluloses during the water leaching. The toluene:ethanol extraction with butylene and propylene oxide has lower EMC values with the solid wood.

Effectiveness of decay resistance was measured in terms of percentage of wood weight loss. The most effective treatments resulted in <5% weight loss; moderately effective treatments, >5% but <30% weight loss; and ineffective treatments, >30% weight loss. The percentage weight loss after exposure to *G.trabeum* of AA, BO, and PO modified southern pine solid wood and fiber is presented in Table 4.

The AA modified solid wood shows moderate effectiveness at 19% weight gain for the unleached (5.0% weight loss) and leached (8.2% weight loss) specimens. At the same time the EMC is significantly lowered from 20.2% to 8.6% for the unleached and from 22.1% to 9.2% for the leached specimens.

The AA modified fiber was effective in arresting decay at 13% weight gain for the unleached (1.4% weight loss) and leached (2.7% weight loss) specimens. The EMC was also lowered from 12.4% to 6.4% for the unleached and from 12.2% to 6.6% for the leached specimens.

The BO modified solid wood shows moderate effectiveness at 23% weight gain for the unleached (7.9% weight loss), leached (8.8% weight loss), and extracted (13.1% weight loss) specimens. The EMC was also lowered from 20.0% to 10.7% for the unleached, from 19.9% to 12.4% for the leached, and from 16.4% to 1 1.7% for the extracted specimens.

The BO modified fiber was effective in arresting decay at 18% weight gain for the unleached (1.0% weight loss), leached (-0.1% weight loss), and extracted (2.3% weight loss) specimens. The EMC was also lowered from 14.4% to 11.3% for the unleached, from 14.6% to 10.9% for the leached, and from 12.5% to 1 1.3% for the extracted specimens.

The PO modified solid wood was ineffective in arresting decay at all levels of weight gain. The EMC was lowered slightly for unleached (from 20.0% to 16.9%), and leached (from 19.9% to 17.3%) specimens. The extracted specimens showed no lowering of the EMC.

The PO modified fiber was effective in arresting decay at 15% weight gain for the unleached (3.2% weight loss), at 21% weight gain for the leached (1.7% weight loss), and at 15% weight gain for the extracted (3.1% weight loss) specimens. The EMC was not lowered for any of the PO modified fiber specimens.

In all cases, except for PO modified fiber, as the EMC is lowered, so is the weight loss due to fungal attack. This indicates that the mechanism of effectiveness is by lowering the cell wall moisture content below the level required for microorganism attack. PO fiber is the exception: the EMC is not lowered with biological efficacy. This indicates that the mechanism of effectiveness may be due to substrate modification.

Overall the AA, BO, and PO modified fiber was more biologically effective at lower percentage weight gains than the solid wood. This is explainable by better accessibility of the hydroxyl sites with the fiber material.

Table 1. Weight percent gain (WPG) and percentage acetyl content of acetylated southern pine solid wood and fiber before and after water leaching.

	Acetylation	Unleached		Water Leached	
	Reaction time	WPG	Acetyl (%)	WPG	Acetyl (%)
Solid Wood	0 min	0.0	1.5	-1.6	1.1
	3 min	5.1	7.3	5.1	6.5
	5 min	11.9	12.1	11.8	12.6
	20 min	14.4	15.0	14.3	14.6
	240 min	18.8	19.5	18.7	19.6
Fiber	0	0	1.0	-3.9	1.0
	22 minutes	4.5	7.5	4.1	6.4
	40 minutes	8.8	11.9	8.6	12.0
	120 minutes	13.4	15.3	12.8	15.5
	240 minutes	14.8	16.3	14.3	17.0

Table 2. Weight percent gain of butylene oxide modified solid wood and fiber before and after water leaching and toluene:ethanol (2:1) extraction.

	Butylene Oxide	Unleached	Tol:Eth Extracted	Water Leached
	Reaction time	(WPG)	(WPG)	(WPG)
Solid Wood	0 min.	0	1.2	-1.3
	75 min.	3.6	2.4	0.0
	2 hours	6.8	5.2	3.9
	4 hours	12.0	9.3	7.4
	6 hours	18.6	15.7	13.9
	6 hr. (120 °C)	23.2	19.9	17.9
Fiber	0	0	-3.3	-2.1
	20 min	6.5	6.1	6.1
	1 hours	11.8	11.1	11.1
	2 hours	17.9	17.0	17.0
	4 hours	20.7	19.6	19.9
	6 hours (120 °C)	20.9	19.5	20.2

Table 3. Weight percent gain of propylene oxide modified solid wood and fiber before and after water leaching and toluene:ethanol (3:1) extraction.

	Propylene Oxide	Unleached	Tol:Eth Extracted	Water Leached
	Reaction time	(WPG)	(WPG)	(WPG)
Solid Wood	0 min.	0	1.2	-1.3
	15 min	6.1	4.3	0.9
	30 min.	12.5	7.9	6.4
	50 min	26.1	20.9	19.4
	75 min	30.1	25.6	21.9
Fiber	0	0	-3.3	-2.1
	5 min	7.5	7.1	7.0
	15 min	15.3	11.7	14.2
	30 min	21.0	19.8	19.6
	60 min	24.9	23.5	23.6
	60 min (120 °C)	25.7	24.4	24.1

Table 4. EMC and weight loss (*G.trabeum*) of chemically modified southern pine solid wood and fiber.*

Chemical Modification	*Solid Wood*				*Fiber*				
	WPG	30% RH	65% RH	90% RH	Wt. Loss (%)	WPG	30% RH	65% RH	90% RH	Wt Loss (%)
Acetic anhydride										51.7
	UL C	4.2	9.4	20.2	65.8	UL C	2.9	7.9	12.4	26.5
	UL 5	2.9	7.0	16.5	58.8	UL 5	2.2	6.2	10.1	5.9
	UL 12	2.4	5.2	13.0	44.9	UL 9	1.6	5.2	8.5	1.4
	UL 14	1.8	4.6	11.4	35.6	UL I3	1.0	3.8	6.4	0.7
	UL 19	1.1	3.7	8.6	5.0	UL I5	0.9	3.5	5.9	55.3
	L C	5.0	11.0	22.1	64.4	LC	2.7	7.7	12.2	34.3
	L 5	4.0	8.9	18.2	52.0	L 5	2.1	6.4	10.4	13.6
	L 12	2.7	6.2	14.1	33.6	L 9	1.5	5.I	8.5	2.7
	L 14	2.4	5.5	12.2	31.1	L 13	1.0	3.9	6.6	0.4
	L 19	1.7	4.3	9.2	8.2	L 15	1.0	3.6	6.1	51.7
Butylene oxide	UL C	5.1	10.9	20.0	65.8	ULC	2.4	7.4	14.4	32.4
	UL 4	3.9	8.6	16.9	62.I	UL 7	2.3	6.8	15.2	11.9
	UL 7	3.4	8.0	15.9	45.4	UL 12	2.0	6.2	14.4	1.0
	UL 12	2.7	6.7	13.8	29.0	UL 18	1.5	5.I	11.3	1.0
	UL 19	2.2	5.8	12.2	23.5	UL21	1.4	4.7	10.3	
	UL 23	1.7	4.9	10.7	7.9	UL.21	1.7	4.7	10.1	0.4
										55.3
	LC	5.3	11.2	19.9	64.4	LC	2.7	7.7	14.6	39.7
	L 4	4.9	9.I	16.8	52.9	L 7	2.9	7.8	15.3	15.7
	L 7	4.3	9.6	17.5	50.0	L 12	2.3	6.7	13.6	-0.1
	L 12	3.7	8.6	16.0	34.6	L 18	I.7	5.3	10.9	-0.2
	L 19	2.7	6.8	13.0	25.2	L21	1.3	4.7	10.1	-0.6
	L 23	2.4	6.2	12.4	8.8	L21	I.4	4.7	9.9	
	E C	4.2	9.0	16.4	69.3	EC	2.4	6.8	12.5	57.3
	E 4	4.1	9.I	16.8	65.9	E 7	2.7	7.5	14.7	41.5
	E 7	3.7	8.4	15.9	52.0	E 12	2.1	6.5	13.2	17.9

	E 12	3.2	7.6	14.7	36.5	E 18	1.6	5.4	11.3	2.3
	E 19	2.5	6.3	12.5	26.3	E 21	1.5	4.8	10.2	2.4
	E 23	2.1	5.6	11.7	13.1	E21	1.5	4.8	10.0	0.2
Propylene oxide	UL C	5.1	10.9	20.0	65.8	ULC	2.4	7.4	14.4	51.7
	UL 6	3.7	8.5	17.4	47.7	UL 8	2.4	7.1	16.3	15.7
	UL 13	3.0	7.5	17.1	43.4	UL 15	2.1	7.0	17.0	3.2
	UL 26	2.2	6.6	16.9	37.7	UL 21	1.9	6.5	16.4	1.6
	UL 30	2.0	6.4	16.9	27.0	UL 25	1.5	5.9	15.5	0.9
						UL 26	1.7	6.1	16.1	1.5
	LC	5.2	11.2	19.9	64.4	LC	2.7	7.7	14.6	55.3
	L 6	4.8	10.5	19.2	47.8	L 8	2.9	7.9	15.6	43.5
	L 13	4.0	9.5	18.4	42.1	L 15	2.8	7.8	15.9	23.8
	L 26	2.9	7.9	17.3	36.2	L21	2.0	6.7	15.2	1.7
	L 30	2.9	7.0	17.3	32.6	L 25	1.7	6.1	14.5	0.2
						L 26	1.6	6.2	14.7	0.4
	E C	4.2	8.9	16.4	69.3	EC	2.4	6.8	12.5	57.3
	E 6	3.9	8.7	17.0	48.2	E 8	2.8	7.7	15.1	26.9
	E 13	3.5	8.3	17.4	45.7	E 15	2.3	7.4	15.3	3.1
	E 26	2.7	7.4	17.3	34.8	E21	2.0	6.7	15.2	-0.5
	E 30	2.5	7.0	17.3	27.9	E25	1.8	6.1	15.0	0.2
						E26	1.7	6.1	14.9	0.5

C = control, UL = unleached, L = water leached, E = toluene:ethanol(2:1) extracted

CONCLUSION

Chemically modified southern pine solid wood and fiber specimens reacted with AA or BO show a correlation between the EMC and fungal resistance. Biological resistance increased with decreasing EMC. This indicates the mechanism of effectiveness is due to lowering of the cell wall moisture content.

Chemically modified southern pine solid wood and fiber specimens reacted with PO did not show a correlation between the EMC and fungal resistance. The EMC was not decreased, yet showed biological resistance with the fiber specimens. This indicates that the mechanism of effectiveness may be due to substrate modification.

Chemical modification of fiber with BO and PO is faster, less water leachable, and less solvent extractable than with the solid wood, even at the slightly lower reaction temperature. These attributes give it great potential as a fiber source for composite materials that will be exposed to adverse environments.

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Problem 2 **Chemical and structural modifications of wood–based materials are required to improve properties and to maximize end use performance of bio–based composite materials.**

FY2001 Research Attainments

Publications

Research Unit

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