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Effect of Aqueous Polymer Treatments on Wood Properties
Part II: Mechanical Properties

by

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Abstract

Partially air-dried sapwood of sweetgum (*Liquidambar styraciflua*) and southern pine (*Pinus* spp.) was treated with either aqueous polyacrylate or aqueous dimethyloldihydroxyethyleneurea (DMDHEU) solutions. Tests for static bending, toughness, and hardness were conducted on matched treated and untreated pieces according to ASTM Standards. Properties of pine were not affected by treatment with the polyacrylate. With sweetgum, the modulus of rupture and modulus of elasticity were reduced, while hardness was improved. For the DMDHEU treatment, reduction in property values for both species was related to curing temperature.

Keywords: Mechanical properties, hardness, toughness, sweetgum, southern pine, polyacrylate, dimethyloldihydroxyethyleneurea, cross-linking, glyoxal, polymer, acrylic

Introduction

Traditional treatments to dimensionally stabilize wood are effective but are too expensive for many potential applications. Treating green or partially dried wood with water soluble polymers may be more cost effective. However, there are no reports on the effect of new polymer systems on the mechanical properties of wood. Strength losses with cross-linking systems have been attributed both to the acid catalyst and to the embrittlement caused by the inflexible formaldehyde crosslinking unit. The use of other crosslinking agents which have longer chain lengths could overcome these problems (Rowell and Youngs 1981).

The objectives of this study were to evaluate: (1) the effects of aqueous polyacrylate and aqueous dimethyloldihydroxyethyleneurea (DMDHEU) treatments on the mechanical properties of wood treated in a partially air-dried condition, and (2) to evaluate the differences in mechanical properties between angiosperm and coniferous species treated with these polymer systems.

Materials and Methods

The materials and treatment procedures used in this study were covered in Part I of this series. Test species were sweetgum (*Liquidambar styraciflua* L.) and southern pine (*Pinus taeda* L. or *P. echinata* Mill.).

Mechanical Testing--The testing scheme for the study is shown in Table 1.

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Defect-free static bending, toughness, and hardness test specimens were cut from each equilibrated sample. Prior to testing, all test specimens were conditioned to 12 percent equilibrium moisture content (EMC). Tests were conducted according to the methods described in ASTM D-143-78 (1982).

Table 1. Treatments for Phases I and II of the study.

Treatment class	Reagent	Catalyst system	Curing type
Phase I			
Control 1	Water	None	Air dry
Control 2	Water	None	Air dry, then heat at 55°C
Control 3	None	None	Air dry
Polyacrylate	6% HA16	None	Air dry
DMDHEU	6% Aerotex 900	0.75% methane sulfonic acid	Air dry, then heat at 55°C
Phase II			
DMDHEU	6% Aerotex 900	0.75% methane sulfonic acid	Air dry, then heat at 55°C
DMDHEU	6% Aerotex 900	0.75% methane sulfonic acid	Air dry, then heat at 80°C

Static Bending--Static bending was performed according to the secondary procedure of ASTM D-143-78 except that the rate of loading was 5 mm/min (0.2 in/min), four times the standard rate. Loading rate was altered to decrease the time required for testing. The tests were carried out on a 13,600 kg Tinius-Olsen universal testing machine. Load-deflection curves were recorded via an x-y plotter. Mechanical properties calculated from the load-deflection curves included modulus of rupture (MOR), modulus of elasticity (MOE), fiber stress at proportional limit (S_{PL}), work to proportional limit (W_{PL}), and work to maximum load (W_{ML}). A software package and sonic digitizer were employed to determine the area under load-deflection curves. At the completion of each test, the failure mode was noted. A section was removed close to the failure to determine moisture content (MC) and specific gravity (green volume basis) (SG) at the time of testing. For treated samples, oven-dry weights were corrected by subtracting the extra weight added by chemical treatment.

Toughness--These tests were carried out according to ASTM D-143-78 (1982). The load was applied to radial and tangential surfaces on alternate specimens. The weight position and initial angle of the pendulum were recorded, and the final angle was read to the nearest 0.1 degree on the vernier attached to the machine. To maintain accuracy, adjusted toughness was calculated on the basis of actual dimensions. Moisture content sections were obtained from near the area of failure.

Hardness--Hardness samples were loaded sequentially to the four sides, and the average side hardness was computed.

Experimental Design--Statistical analyses were performed on mechanical property values to detect any changes in the treated material compared to the control groups. To evaluate the mechanical properties, a completely randomized design with three levels of controls was first analyzed for each species independently. To correct for expected differences in mechanical properties, MC and SG were chosen as concomitant variables, and a covariance analysis was performed on the data. Adjusted treatment means were separated by using Tukey's test.

The correlation of the two covariates and the mechanical property values was tested using an F-test. In the case of nonsignificant correlation, an ordinary analysis of variance was performed. Controls not significantly different from each other were pooled as one treatment level and analysis of covariance or analysis of variance was again executed to detect differences between polymer treatments and control(s).

In Phase II, a separate analysis of covariance was used to detect any propoerty changes compared to control(s), and differences between the high and low curing temperatures. Because the samples in this analysis were not matched, the mechanical properties were adjusted for differences in MC and SG at the time of test.

Results and Discussion

Analysis of Mechanical Properties for Control Groups--Control groups were analyzed to determine if any differences occurred due to either the treating process or heating process. Mechanical property data were adjusted for differences in MC and SG. The adjusted mean property values for small clear specimens of sweetgum and southern pine control groups are presented in Table 2. Group means with the same letter are not significantly different. Unequal sample sizes were used due to the limited clear wood test specimens that could be obtained from each sample.

Except for MOR, S_{PL}, and W_{PL}, the mechanical properties did not significantly differ among the three sweetgum control groups. The MOR, S_{PL}, and W_{PL} of the two water-treated control groups were significantly reduced when compared to the untreated group. However, the kiln-heated and air-dried water-treated groups were not significantly different for any property. Therefore, these two groups were combined for subsequent comparisons. The radial and tangential toughness were pooled as one treatment level since they were not significantly different. Mechanical properties among southern pine control groups did not differ significantly and they were pooled as one treatment level in subsequent analyses.

Effect of Polymer Treatments on Mechanical Properties--The adjusted means for each treatment group are presented in Table 3. The values in parentheses represent the change in mechanical properties compared to either the untreated controls or the pooled control value.

Static bending--A significant reduction of 6.8 percent was found for the average MOE values of polyacrylate-treated sweetgum. No difference in MOE was found between the DMDHEU-treated material and the controls. The MOR values for both polymer treatments were significantly lower than the untreated control, but they did not significantly differ from the water-treated control group. This result suggests that the impregnation alone adversely affects the

MOR of sweetgum. The average MOR was reduced by 11.4 percent when treated with polyacrylate and by 11.8 percent when treated with DMDHEU. Nonsignificant differences were found for S_{PL}, W_{PL}, and W_{ML} for both polymer treatments when compared with the untreated control group.

Table 2. Adjusted mean property values for small, clear specimens of control groups.[1]

Property	Water-treated[2] KHAT	Water-treated[2] ADAT	Untreated	Averages[3] Water-treated	Averages[3] All
			Sweetgum		
Static Bending					
MOE (MPa)	9,412 A	9,405 A	10,508 A	9,408	9,792
MOR (kPa)	76,217 B	71,349 B	90,524 A	73,610	
S_{PL} (kPa)	46,141 B	41,549 B	55,277 A	43,681	
W_{PL} (kJ/m^3)	6.0 B	5.5 B	8.3 A	6.6	
W_{ML} (kJ/m^3)	114.6 A	106.9 A	138.6 A	110.5	120.3
Toughness (J)[4]	38.6 A	35.7 A	42.5 A	37.2	39.0
Hardness (N)	4,733 A	4,715 A	5,266 A	4,723	4,910
			Southern pine		
Static Bending					
MOE (MPa)	5,447 A	5,564 A	5,323 A	5,510	5,445
MOR (kPa)	81,968 A	83,609 A	83,685 A	82,847	83,139
S_{PL} (kPa)	46,003 A	51,933 A	49,334 A	49,180	49,234
W_{PL} (kJ/m^3)	13.2 A	11.1 A	12.5 A	12.1	12.2
W_{ML} (kJ/m^3)	154.9 A	140.7 A	154.9 A	147.3	150.0
Toughness (J)					
Radial	21.0 A	24.1 A	23.0 A	22.8	22.9
Tangential	26.7 A	28.9 A	27.9 A	27.9	27.9
Hardness (N)	3,496 A	3,461 A	3,630 A	3,477	3,525

[1] Means followed by the same letter are not significantly different (α = 0.05) using Tukey's test.
[2] KHAT = Air-dried after treatment followed by kiln heating; ADAT = Air-dried after treatment.
[3] Averages computed for means not significantly different; means not significantly different were pooled for subsequent comparisons.

Table 3. Adjusted mean property values for small, clear specimens removed from HA16-treated and DMDHEU-treated dimension stock compared with control groups.[1]

Property	HA16	DMDHEU	Controls[2]		
			Untreated	Water-treated	All
Sweetgum					
STATIC BENDING					
MOE (MPa)	9,122 B	9,508 A			9,792 A
Reduction (%)[3]	-6.8	-2.9			
MOR (kPa)	80,223 B	79,858 B	90,524 A	73,610 B	
Reduction (%)	-11.4	-11.8		-18.7	
S_{PL} (kPa)	46,665 A	49,058 A	55,277 A	43,681 A	
Reduction (%)	-15.6	-11.3		-21.0	
W_{PL} (kJ/m^3)	6.9 A	7.2 A	8.3 A	6.6 A	
Reduction (%)	-16.9	-12.8		-20.5	
W_{ML} (kJ/m^3)	129.3 A	110.5 A			120.3 A
Reduction (%)	7.5	-8.2			
TOUGHNESS					
Toughness (J)[4]	41.8 A	38.0 A			39.0 A
Reduction (%)	7.2	-2.7			
HARDNESS					
Hardness (N)	5,311 A	4,790 B			4,910 B
Reduction (%)	8.2	-2.4			
Southern pine					
STATIC BENDING					
MOE (MPa)	5,764 A	5,523 A			5,445 A
Reduction (%)	5.9	1.4			
MOR (kPa)	85,574 A	83,567 A			83,138 A
Reduction (%)	2.9	0.5			
S_{PL} (kPa)	49,954 A	49,968 A			49,234 A
Reduction (%)	1.5	1.5			
W_{PL} (kJ/m^3)	11.7 A	13.0 A			12.2 A
Reduction (%)	-4.0	6.8			
W_{ML} (kJ/m^3)	147.6 A	133.6 A			150.0 A
Reduction (%)	-1.6	-10.9			
TOUGHNESS					
Radial (J)	22.1 A	20.3 B			22.9 A
Reduction (%)	-3.3	-11.2			
Tangential (J)	31.5 A	24.9 B			27.9 A
Reduction (%)	13.0	-10.9			
HARDNESS					
Hardness (N)	3,607 A	3,487 A			3,525 A
Reduction (%)	2.3	-1.1			

[1] Means followed by the same letter are not significantly different ($\alpha = 0.05$) using Tukey's test.

[2] The two water-treated control groups were not significatly different ($\alpha = 0.05$) using Tukey's test, and were pooled to give average values. If a value for All is given, none of the controls were significantly different.

[3] Compared to untreated or average of all controls.

With southern pine, the MOE, MOR, and S_{PL} values of both treatments were higher than the control group values, but this increment was not significant. W_{PL} and W_{ML} of polyacrylate-treated and DMDHEU-treated material did not significantly differ from the control group, although a 6.8 percent improvement for W_{PL} occurred for DMDHEU-treated southern pine. Most of the bending samples failed normally, first in compression followed by simple tension. Most of the treated sweetgum samples exhibited compression failure at low load levels followed by a tension failure at large deformations. Probably, the presence of polymer (which either crosslinked with the cellulose or bulked the wood cell wall) in sweetgum reduced the lateral support of the wood fibers and increased their buckling under the compressive load. In treated pine, stiffness was unaffected and the wood deformed in a manner identical to the untreated controls. Reduction in strength properties is likely too small to be of any practical consequence.

Toughness--Since radial and tangential toughness values for the treated sweetgum were not significantly different, values were pooled for analysis. Neither polyacrylate-treated material nor DMDHEU-treated material was significantly different from untreated controls, although polyacrylate treatment improved toughness 7.2 percent. A 13 percent increase in tangential toughness of southern pine treated with polyacrylate was observed, but this increment was not significant. The DMDHEU treatment reduced both the radial and tangential toughness of southern pine.

Hardness--The polyacrylate treatment significantly increased the hardness of treated sweetgum by 8.2 percent, but hardness was not affected by DMDHEU treatment. The hardness of southern pine was virtually unaffected by either treatment.

Effect of High Temperature Curing on Mechanical Properties of DMDHEU-treated Wood--An analysis of covariance was used to detect any property changes between the high and low curing temperatures for DMDHEU-treated material compared to the control group(s). The adjusted mean properties of small clear specimens are summarized in Tables 4.

Static bending--The MOE and W_{ML} of the treated sweetgum did not differ significantly among groups. Significant reductions were found for the MOR, S_{PL}, and W_{PL} of treated sweetgum cured at 80°C when compared either to control groups or to treated groups cured at 55°C. With southern pine cured at 80°C, the MOE, MOR, S_{PL}, and W_{PL} were significantly different from controls. No significant change in these properties was observed for the low-temperature cured groups, and W_{ML} was not significantly reduced by either curing temperature.

In the static bending test, the treated material cured at 80°C was crushed at the point of contact with the loading fixture, with final failure occurring in tension. The crushing was noted at low load levels and was more severe than that of treated material cured at 55°C.

Toughness -Toughness was not significantly different among the three sweetgum groups. The two curing temperatures did not significantly differ in their effect on radial or tangential toughness of treated southern pine. When compared to the untreated control group, the radial and tangential toughness of high-temperature cured southern pine were reduced 11.2 and 10.9 percent, respectively.

Table 4. Adjusted mean property values for small, clear samples for DMDHEU-treated wood cured at 55° and 80°C compared with control groups.[1]

Property	Cure Temperatures 55°C	80°C	Controls Untreated	Water-treated[2]	All
		Sweetgum			
STATIC BENDING					
MOE (MPa)	9,667 A	8,977 A			9,792 A
Reduction (%)[3]	-1.3	-8.3			
MOR (kPa)	79,293 B	70,660 C	90,524 A	73,610 B	
Reduction (%)	-12.4	-21.9		-18.7	
S_{PL} (kPa)	49,796 A	37,509 B	55,277 A	43,681 A	
Reduction (%)	-9.9	-32.1		-21.0	
W_{PL} (kJ/m^3)	7.3 A	4.3 B	8.3 A	6.6 A	
Reduction (%)	-11.9	-47.7		-20.5	
W_{ML} (kJ/m^3)	104.0 A	117.8 A			120.3 A
Reduction (%)	-13.5	-2.1			
TOUGHNESS					
Toughness (J)	38.0 A	40.4 A			39.0 A
Reduction (%)	-2.7	3.7			
HARDNESS					
Hardness (N)	4,595 A	4,355 B			4,910 A
Reduction (%)	-6.4	-11.3			
		Southern pine			
STATIC BENDING					
MOE (MPa)	5,509 A	4,695 B			5,445 A
Reduction (%)	1.2	-13.8			
MOR (kPa)	84,691 A	70,129 B			83,138 A
Reduction (%)	1.9	-15.6			
S_{PL} (kPa)	50,423 A	39,922 B			49,234 A
Reduction (%)	2.4	-18.9			
W_{PL} (kJ/m^3)	13.0 A	9.2 B			12.2 A
Reduction (%)	6.8	-24.8			
W_{ML} (kJ/m^3)	135.4 A	131.0 A			150.0 A
Reduction (%)	-9.7	-12.7			
TOUGHNESS					
Radial (J)	20.9 A	22.1 A			22.9 B
Reduction (%)	-8.7	-3.3			
Tangential (J)	24.9 A	25.4 A			27.9 B
Reduction (%)	-10.9	-8.9			
HARDNESS					
Hardness (N)	3,536 A	3,772 A			3,525 A
Reduction (%)	0.3	7.0			

[1] Means followed by the same letter are not significantly different (α = 0.05) using Tukey's test.
[2] The two water-treated control groups were not significatly different (α = 0.05) using Tukey's test, and were pooled to give average values. If a value for All is given, none of the controls were significantly different.
[3] Compared to untreated or average of all controls.

Hardness--Hardness tests showed no significant differences among the three group means for southern pine, but a significant 11.3 percent reduction was observed for sweetgum cured at 80°C.

Possible Mechanisms for Losses Caused by Polymer Treatment--The presence of an acid catalyst and heat to initiate crosslinking will effectively rupture microfibrils thus creating shorter cellulose chains. Since most mechanical properties of wood are closely related to cellulose microfibril strength, a treatment which reduces the microfibril integrity will also reduce the toughness and bending strength (Ifju 1964). Embrittlement of wood treated with a short inflexible crosslinking unit of the O-C-O type has been reported (Stamm 1959, Tarkow and Stamm 1953). The use of a longer chain length crosslinking agent, such as the DMDHEU, is likely to form more flexible crosslinks and reduce the embrittlement of wood.

In the present study, except for MOE in sweetgum and toughness in southern pine, mechanical properties were unaffected at the 55°C curing temperature (see Table 3). However, higher curing temperatures caused significant reductions in most mechanical property values of both species (Table 4). The reduction in MOE and MOR of pine cured at high temperature (90°-120°C) was also reported by Nicholas and Williams (1987). A possible cause of this strength reduction is the reaction of hemicellulose acetyl groups to form acetic acid. This acid depolymerizes cellulose microfibrils located in the amorphous regions, which creates shorter chains of cellulose (Hillis 1975). Treatment with DMDHEU solution (containing an acid catalyst) followed by drying at elevated temperature accelerates the rate of strength loss.

The reduction of MOE and MOR with the introduction of polyacrylate into sweetgum may be attributed to cell wall bulking. Due to the swelling action of the polymer, a cross-section of treated wood contains fewer fibrils of cellulose than an untreated, dry section; therefore, the strength of the treated wood is reduced. Additionally, comparison of the failure modes of treated and untreated samples suggests that treatment produces a weakening that leads to buckling. Published data are not available to verify these mechanisms, and more research is needed to support these concepts. Most polyacrylate-treated southern pine static bending samples failed in a manner identical to the controls, which suggests that treatment had little effect.

Summary and Conclusions

This study shows that some clear wood mechanical properties are affected by polymer treatment. The polyacrylate treatment did not affect any property of southern pine. For sweetgum, however, MOE and MOR were reduced while hardness was improved. The DMDHEU treatment followed by curing at 55°C significantly reduced both radial and tangential toughness of southern pine and reduced the MOR of sweetgum. When compared to untreated controls, the MOR, S_{PL}, and W_{PL} of both DMDHEU-treated species cured at 80°C were significantly reduced. High-temperature curing also reduced radial and tangential toughness and MOE of treated southern pine and reduced hardness of treated sweetgum.

The reduction in mechanical property values of DMDHEU-treated wood may be due to acetic acid production and subsequent depolymerization of the cellulose chains. Curing at higher temperatures should exacerbate this degradation. Losses in strength and stiffness with polyacrylate-treated wood may be due to bulking of the wood cell wall.

The property reductions observed in this study for treated wood do not, in general, represent a serious detriment to use. For structural applications, no reduction in design values appears necessary for southern pine.

Literature Cited

Annual Book of American Society for Testing and Materials (1982) Standard methods of testing small clear specimens of timber, ASTM D143-78, Philadelphia, PA.

Frick, J.G. Jr. (1971) Current perspective of durable press. Chem. Tech. 2:100-107.

Hillis, W.E. (1975) The role of wood characteristics in high temperature drying. J. Inst. Wood Sc. 7(2):60-67.

Ifju, G. (1964) Tensile strength behavior as a function of cellulose in wood. Forest Prod. J. 14(8):366-372.

Nicholas, D.D., and A.D. Williams (1987) Dimensional stabilization of wood with dimethylol compounds. IRG/WP/3412.

Parikh, D.V., and R.M. Reinhardt (1984) Low energy cure for low H_2CO release DP fabrics. Am. Dyestuff Reporter (March), p. 44-50.

Rowell, R.M., and R.L. Youngs (1981) Dimensional stabilization of wood in use. U.S. For. Serv., For. Prod. Lab. Res. Note FPL-0243. For. Prod. Lab. Madison, Wis.

Stamm, A.J. (1959) Dimensional stabilization of wood by thermal reactions and crosslinking. Tappi 42(1):39-44.

Tarkow, H., and A.J. Stamm (1953) Effect of formaldehyde treatments upon the dimensional stabilization of wood. J. For. Prod. Res. Soc. 3(3):33-37.