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Effect of Heat Treatment on the Dimensional Stability and the Bending Properties of Radiata Pine Sapwood¹

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ABSTRACT

The effects of heat treatment on the dimensional stability and bending properties of radiata pine sapwood were investigated. The dimensional stability was almost achieved by heat treatment though the loss of strength was accompanied as a negative effect. The improvement in dimensional stability of wood and the resultant reduction in bending properties were closely related to treatment temperature and duration. The optimum treatment conditions, which could be used to achieve a desired improvement in dimensional stability with resultant losses in modulus of rupture were proposed based on the results obtained in this study.

Key words: heat treatment, radiata pine, dimensional stability, bending property

INTRODUCTION

Wood modification by non-chemical process is needed to completely exclude the environmental impact related to the use of chemicals for improving decay resistance and dimensional stability. One feasible non-chemical method is heat treatment at high temperatures. The rectification process, that is, heat treatment in the absence of oxygen, has shown that biodegradation resistance and dimensional stability could be improved to varying extent according to heating temperature and duration (Dirol and Guyonnet, 1993; Troya and Navarrete, 1994). However, the rectification process appears to be less practical since the treatment has to be conducted in the absence of oxygen. It would be preferable if a

treatment in atmospheric conditions could be commercialization. Viitanen *et al.* (1994) have reported promising results from such a preliminary test with spruce. Although decay resistance and dimensional stability can be improved by heat treatment, a simultaneous reduction in mechanical properties of treated wood offset the improvement in properties. Consequently, accurate process control with a well-defined temperature-time schedule is required.

Effect of heat treatment on the biodegradation resistance and dimensional stability of radiata pine sapwood was explored. In this report, we discuss the effects of heat treatment on the dimensional stability and the bending properties of wood. The optimum treatment conditions to achieve comparable dimensional stability to that

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obtained by traditional polyethylene glycol (PEG) treatment is identified, while minimizing strength losses. In previous paper, we reported effects of heat treatment on the decay resistance (Kim *et al.*, in press).

MATERIALS and METHODS

Specimen preparation

A total of 930 bending specimens (15 mm x 20 mm x 280 mm long) were prepared from freshly sawn radiata pine (*Pinus radiata* D. Don) sapwood lumber, and then divided into 31 groups (one control group and 30 heat treatment groups) of 30 specimen each, with each group having similar distributions of green weight and percent latewood. The thirty treatment groups consisted of combinations of two initial moisture conditions (green or air-dried), three heating temperatures (120, 150, or 180C), and five heating periods. After grouping, half of the material in each heat treatment group was air-dried until the moisture content was about 15%.

Heat treatments

Each treatment group was heated at a given temperature for a given period in a convection oven. The heating period at 120, 150, and 180° C was 12, 24, 48, 72, or 96 hours, 6, 12, 24, 36, or 48 hours, and 4, 8, 12, 16, or 20 hours, respectively. Following heat treatment, all specimens including untreated controls were stacked indoors until constant weights were obtained. Specimens were then planed to final dimensions of 10 mm x 15 mm x 280 mm long.

Static bending tests

Static bending tests were conducted on a universal testing machine using a center point loading as described in American Society of Testing and Materials Standard (ASTM) D-143 with minor revisions (ASTM, 1994). The span-

to-depth ratio was 14 to 1, with a test span of 210 mm. The load was continuously applied at a rate of 0.75mm/min. Modulus of rupture (MOR), modulus of elasticity (MOE), and work to maximum load (WML) of each specimen were automatically calculated and recorded by data acquisition system connected to the testing machine. Upon completion of bending tests, a 15-mm long block was cut near the area of failure to determine moisture content and specific gravity. Specific gravity was determined using volume at test time and oven-dry weight.

Evaluation of dimensional stability

Following bending tests, a sample measuring $10~\text{mm} \times 15~\text{mm} \times 50~\text{mm}$ was cut from each specimen. Among 30 samples prepared from each group, ten samples having a similar specific gravity and moisture content were selected and used for dimensional stability tests. These samples were oven dried for 24 hours, and then their dimension and weight were measured. They were then conditioned for four weeks under $75\pm2\%$ of relative humidity provided by saturated sodium chloride (NaCl) solution kept at $25\pm2\%$. The dimensions of swollen samples were measured at the same point as the initial dimensions had been measured, and sample weight were measured again.

Dimensional stability was evaluated in terms of volumetric swelling coefficient (S) and water absorption (M), which were calculated using the following equations:

$$S(\%) = \frac{V_s - V_d}{V_d} \times 100$$
 [1]

$$M(\%) = \frac{W_s - W_d}{W_d} \times 100$$
 [2]

where V_s is the volume of swollen sample (cm³), V_d is the volume of oven-dried sample (cm³), W_s is the weight of swollen sample (g), and W_d is the weight of oven-dried sample (g)

The improved dimensional stability of heat-treated wood samples was defined as perecent decrease in volumetric swelling cofficeient(DS) and decrease in water absorption(DM) compared to those of untreated controls, and expressed by following equations (3) and (4).

$$DS(\%) = \frac{S_u - S_t}{S_u} \times 100$$
 [3]

$$DM(\%) = \frac{M_u - M_t}{M_u} \times 100$$
 [4]

where S_u is volumetric swelling cofficeient of untreated sample, S_t is volumetric swelling cofficeient of heat-treated sample, M_u is water absorption of untreated sample, and M_t is water absorption of heat-treated sample.

To compare the improved dimensional stability imparted by heat treatment with that by PEG treatment, ten samples were treated with 75% PEG-1000 solution by vacuum impregnation.

Construction of prediction models

A prediction model for assessing the improved dimensional stability (i.e., a decrease in volumetric swelling coefficient and water absortion) and residual MOR of heat treated wood was developed to optimize improvement in dimensional stability with minimum losses in bending strength.

The relationships of heating period with a decrease in volumetric swelling coefficient (DS) and water absorption (DM) for any heating temperatures were fitted by equation (5).

$$DS(\%)$$
 or $DM(\%) = A_0 + A_1 \times \sqrt{HP}$ [5]

where HP is the heating period (hours), and A_{θ} and A_{I} are constants.

Two nonlinear equations (6) and (7) were tested to find the best for the relations between heating period and losses in bending strength. The nonlinear least squares and nonlinear

regression methods were used to estimate parameters in equations (6) and (7), respectively.

$$MOR = B_2 - B_0[1 - e^{B_1HP}]$$
 [6]

$$MOR = A_1 e^{A_2HP}$$
 [7]

where MOR is bending strength, HP is the heating period (hours), and A_1 , A_2 , B_0 , B_1 and B_2 are constants

RESULTS and DISCUSSION

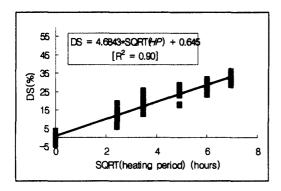
Percent improvement of dimensional stability and residual bending properties of heat treated radiata pine sapwood samples were summarized in Table 1. Heat treatment improved dimensional stability, and the degree of an improvement was increased with increasing both heating temperature and heating period. The increase in dimensional stability was also influenced by initial moisture conditions of wood being heated. Dimensional stability of heated air-dried wood was somewhat greater than that of heated green wood. When compared with PEG-treated groups, the degree of inprovement in dimensional stability for some treatment groups heated at higher temperature and longer period were greater. This means that heat treatment can improve dimensional stability comparable to that achieved by PEG treatment. Although chemical changes of heat-treated wood were not analyzed, it is believed that an improvement in dimensional stability by heat treatment is primarily associated with the permanently reduced hygroscopicity due to a thermal decomposition of hemicelluloses. Stamm (1964) explained that hemicelluloses are thermally decomposed to furfural polymers of various breakdown sugars, which are less hygroscopic than the hemicelluloses from which they are formed. Dirol and Guyonnet (1993) reported that loss of water of constitution, together with thermal decomposition

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Table 1. Effect of heat treatment on the improvement of dimensional stability and bending properties of radiata pine sapwood

Moisture condition	Heating temperature	Heating period	Improved dimensional stability*1		Residual bending property		
			DS	DM	MOR	МОЕ	WML
	(°C)	(Hours)			(%)		
	120	12	6.3	14.0	98.9	93.2	96.8
		24	11.7	20.9	98.6	93.1	95.1
		48	14.3	26.4	98.4	92.5	94.9
		72	18.9	29.5	94.6	92.2	90.1
		96	19.3	34.5	93.9	90.8	83.4
	150	6	9.8	12.7	94.7	93.2	93.6
Dry		12	14.9	19.4	96.1	92.6	82.3
		24	20.1	24.0	91.1	92.3	75.9
		36	22.5	28.0	90.9	91.4	74.0
		48	24.9	32.0	91.1	93.0	66.1
	180	6	15.2	14.9	92.1	95.3	62.7
		8	21.9	26.2	84.4	94.5	53.1
		12	24.4	31.2	79.4	93.9	41.4
		16	26.5	34.6	72.3	92.4	33.6
		20	29.9	40.4	72.1	91.7	32.7
	120	12	5.2	5.8	89.2	94.6	73.9
Green		24	7.3	10.2	89.0	89.9	73.9
		48	11.9	15.6	88.2	89.8	64.9
		72	16.9	17.1	85.5	89.2	59.5
		96	17.5	24.3	79.2	79.8	58.9
	150	6	9.5	9.1	89.2	92.7	65.6
		12	12.8	16.8	85.9	89.9	65.5
		24	16.6	20.9	82.0	88.2	64.8
		36	18.9	23.8	81.4	87.5	54.6
		48	21.0	27.7	79.0	89.3	45.8
	180	6	13.3	9.9	88.7	90.4	82.6
		8	16.9	17.8	81.2	89.9	49.7
		12	22.1	21.9	73.6	88.8	36.0
		16	26.0	25.8	72.0	86.3	34.0
		20	27.8	29.1	68.5	86.8	30.8

The improved dimensional stability of heat-treated wood samples is calculated based on the dimensional stability of untreated controls(0.0%). The DS and DM of 75% PEG-1000 treated samples were 21.4 and 24.7%, respectively.



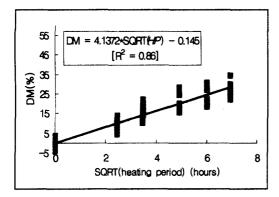


Fig. 1. Relationship between square root of heating period and decrease in volumetric swelling coefficient (DS) and decrease in water absorption (DM) of green samples heated at 150°C.

of hemicelluloses, was also attributed to the improvement in dimensional stability.

Heat treatment decreased bending properties, and the rates of their reduction were related closely to the heating conditions. All bending properties reduced as both heating temperature and duration increased with WML being reduced most severely, while MOE showed a much more moderate rate of reduction (Table 1). Reduction in bending properties was more severe when green specimens were heated. It has been proposed that during heating the carbohydrates are hydrolyzed by acetic acid liberated from the acetyl group of the surrounding hemicelluloses. The resulting strength loss due to the depoly-

merization of the carbohydrate fraction commences with the zone of weakness between the S1 and S₂ layers of the cell wall (Hillis, 1975). Hemicelluloses are attacked more easily than cellulose. mainly because of their amorphous state and relatively low degree of polymerization. Most of their glycosidic bonds are also more labile toward acid hydrolysis than glucosidic bonds in cellulose (Sjöström, 1981). Hillis (1975) also reported that elevated temperature and high moisture contents accelerated production of acetic acid, and thus increased the rate of strength loss. Winandy and Morrell (1993) reported that degradation and/or destruction of hemicelluloses played an important role in incipient loss of wood strength.

The degree of improved dimensional stability at specific temperatures could be predicted using the relationships of heating period with the decrease in both volumetric swelling coefficient and water absorption, fitted by equation (5). Figure 1 presents a graphical depiction of the relation between heating period and improved dimensional stability when improved dimensional stability data are fitted with equation (5). The decrease in both volumetric swelling coefficient and water absorption correlated closely with the heating duration for each temperature employed (Table 2). The relationship between the residual bending properties and heating period was curvilinear. Nonlinear equations (6) and (7) were applied to find a better model described the property loss by heat treatment. Equation (6) describes a reduction limit that is asymptotically approached with the increase in heating period. Although this form of equation describes the reduction in properties very well for a relatively short heating period, extrapolation to longer heating periods may result in inaccurate predictions and hence is not recommended. Consequently, equation (7) was used, even though the initial rapid loss in property was not fitted well, compared to equation (6). The nonlinear regression equations for predicting the residual bending

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Table 2. Results of regression analyses between heating period and decrease in volumetric swelling coefficient and water absorption using Equation (5)*1

Property	Treatment	Regression equation	R ²
	Air-dried samples		
	120℃	DS = 0.1176 + 2.0797*SQRT(HP)	0.86
	150℃	DS = 0.9709 + 3.6379*SQRT(HP)	0.91
Volumetric	180℃	DS = 1.1302 + 6.6221*SQRT(HP)	0.89
swelling coefficient	Green samples		
COCITICICIA	120℃	DS = -0.8501 + 1.9009*SQRT(HP)	0.87
	150℃	DS = 1.2890 + 2.9894*SQRT(HP)	0.87
	180℃	DS = 0.0735 + 6.3002*SQRT(HP)	0.91
	Air-dried samples		
	120℃	DM = 0.9231 + 3.5331*SQRT(HP)	0.89
	150℃	DM = 0.6450 + 4.6843*SQRT(HP)	0.90
Water	180℃	$DM \approx -1.5932 + 9.2892*SQRT(HP)$	0.91
absorption	Green samples		
	120℃	DM = -1.8415 + 2.4711*SQRT(HP)	0.84
	150℃	DM = -0.1456 + 4.1372*SQRT(HP)	0.86
	180℃	DM = -1.8860 + 6.8518*SQRT(HP)	0.86

^{*1}DS, DM, and HP represent decrease in volumetric swelling coefficient, decrease in water absorption, and heating periods (hour), respectively.

Table 3. Results of regression analyses between heating period and MOR using Equation (7)*1

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Treatment	Regression equation	R²			
Air-dried samples					
120℃	$MOR = 1178.06e^{-0.0007(HP)}$	0.91			
150℃	$MOR = 1148.13e^{-0.0018(HP)}$	0.79			
180℃	$MOR = 1164.20e^{-0.0178(HP)}$	0.98			
Green samples					
120℃	$MOR = 1123.85e^{-0.0019(HP)}$	0.79			
150℃	$MOR = 1107.70e^{-0.0043(HP)}$	0.78			
180℃	$MOR = 1143.69e^{-0.0196(HP)}$	0.95			

^{*} MOR and HP represent residual modulus of rupture and heating period (hours), respectively.

strength with correlation coefficients were shown in Table 3. The loss in MOR as described by the nonlinear relationship of equation (7) is shownin Figure 2.

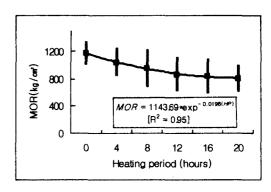


Fig. 2. Relationship between heating period and modulus of rupture of green samples heated at 180°C. The results of equation (7) are represented with the solid line. Vertical bars and solid squares indicate standard deviations and sample means, respectively.

Table 4. Estimated heating periods required to acheive an increase in dimensional stability comparable to that of 75% PEG-treated samples and estimated percent residual MOR at the specific heating periods

Property	Treatment	Heating period	Residual MOR	
		(hours)	(%)	
	Air-dried samples			
	120℃	104	93.1	
	150℃	31	92.2	
Volumetric	180℃	9	83.8	
swelling	Green samples			
coefficient	120℃	117	76.6	
	150℃	45	77.6	
	180℃	11	77.7	
	Air-dried samples			
	120℃	45	97.0	
	150℃	26	93.1	
Water	180℃	6	88.7	
absorption	Green samples			
	120℃	86	81.2	
	150℃	35	80.9	
	180℃	11	78.2	

As discussed above, heating period needed to achieve dimensional stability comparable to that obtained by PEG treatments could be estimated for a given initial moisture conditions of wood and heating temperature using equation (5), and the magnitude of MOR loss resulted from heat treatment could be predicted by equation (7) at given heating conditions and wood moisture conditions. For example, volumetric swelling coefficient and water absorption of air-dried wood heated at 150°C for about 31 and 26 hours were comparable to that by 75% PEG treatment. MOR loss at these conditions can be predicted as about 7.8 and 6.9%, respectively (Table 4).

CONCLUSION

Heat treatment at high temperatures may be a promising approach for improving dimensional stability of wood as a non-conventional method, where strength requirement and aesthetic quality are not important. However, the negative effects of heat treatment, such as loss of strength and discoloration of wood, has complicated the use of heat treatment. Optimum heating conditions, which can be used to achieve a desired improvement in dimensional stability with minimum strength loss, could be derived using the models developed. Further studies are needed to develop well-defined and more confident temperaturetime schedules for heat treatment.

REFERENCES

- American Society for Testing and Materials. 1994.
 The annual book of ASTM standards. Philadelphia,
 PA.
- Dirol, D. and Guyonnet, R. 1993. The improvement of wood durability by rectification process. International Research Group on Wood Preservation, Document No., IRG/WP/93-40015. Stockholm, Sweden.
- Hillis, W. E. 1975. The role of wood characteristics in high-temperature drying. J. Inst. Wood Sci. 7(2): 60-67.
- Kim, G.-H., K.-E. Yun, and J.-J. Kim. 1999. Effect of heat treatment on the decay resistance and the bending properties of radiata pine sapwood. *Material und Organismen* (in press).
- Sjöström, E. 1981. Wood chemistry: fundamentals and applications. Academic Press, Inc. San Diego, CA.
- Stamm, A. J. 1964. Wood and cellulose science.
 The Ronald Press Co. New York, NY.
- Troya, M. T. and Navarrete. M. 1994. Study of the degradation of rectified wood through ultrasonic and gravimetric techniques. The International

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- Research Group on Wood Preservation. Document No: IRG/WP/94-40030.
- Viitanen, H., Jamsa, S., Paajanen, L., Nurmi, A. and Viitaniemi, P. 1994. The effect of heat treatment on the properties of spruce: a preliminary report. The International Research Group on Wood
- Preservation. Document No: IRG/WP/94-40032. Stockholm, Sweden.
- Winandy, J. E. and Morrell, J. J. 1993. Relationship between incipient decay, strength, and chemical composition of douglas-fir heartwood. Wood & Fiber Sci. 25(3): 278~288.