

Effects of Heating Temperature and Time on the Mechanical Properties of Heat-Treated Woods¹

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ABSTRACT

This study was performed to investigate the effects of heat treatment on the mechanical properties of two species of wood under different heating conditions including at 180°C for 12 h and 24 h, and at 210°C for 3 h and 6 h. Two species of wood, *Pinus densiflora* and *Larix kaempferi*, were exposed to different heat treatments to assess the effects on the volume change, bending properties in static and dynamic mode and compressive strength. The results showed heat treatment caused significant changes in mechanical properties such as the static and dynamic moduli of elasticity (MOE_d and MOE_s), and the modulus of rupture (MOR). The volume of the wood after heat treatment decreased as the heating temperature and time were increased. The bending strength performance of the wood after heat treatment decreased as the heating temperature and time were increased. The effect of heat treatment at a high temperature on the bending MOR was greater in both species than that for a long time. However, the compressive strengths of all the heat-treated samples were higher than the control sample. Furthermore, highly significant correlations between MOE_d and MOR, and MOE_s and MOR were found for all heating conditions.

Keywords : heat-treated wood, *Pinus densiflora*, *Larix kaempferi*, volume, bending and compressive strength properties

1. INTRODUCTION

Heat treatment of wood is an environmentally friendly wood preservation technique. Wood is heated to temperatures above 200°C depending on the wood species and the required wood properties (Kocaeffe *et al.* 2010). This thermal treatment is an alternative to chemical treatment

for wood preservation, which has sometimes been used to improve timber quality (Awoyemi and Jones. 2011; Poncsak *et al.* 2006). Heat treatment has been researched throughout the world as a method for removing resin from conifer, changing of the material qualities of softwood and hardwood, and improving dimensional stability (Shin *et al.* 2009). Heat-treated

¹ Received on December 24, 2014

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wood is more dimensionally stable under changing humidity, and the mechanical properties of heat-treated wood show higher performance (Kim *et al.* 2009). Relatively mild thermal treatments of wood in a two-step process, have been shown to improve dimensional stability (Tjeerdsma *et al.* 1998), and sound absorption properties were also improved by heat treatment (Byeon *et al.* 2010). Heat treatment has also been reported to improve the decay resistance of some wood species; the improvement was attributed to reductions in hemicellulose content, moisture, and other wood components such as starch, fatty acids, and lipids which are essential for mold and fungi growth (Kocaefe *et al.* 2008).

However, heat treatment can also weaken the mechanical properties of wood. The degradation of hemicellulose that connects cellulose and lignin in the cell wall causes deterioration in strength. Deterioration in toughness, hardness, bending, compression, and tensile strength due to heat treatment have also been reported (Yildiz *et al.* 2002). Changes in the chemical structure of wood during thermal treatment could be explained by a modification of conformational arrangement of wood biopolymers due to a loss of residual water, or more probably to the plasticization of lignin (Sivonen *et al.* 2002; Hakkou *et al.* 2005). A decrease in hydroxyl groups increases the hydrophobicity of wood and reduces the absorption of water (Kamdem *et al.* 2002; Stamm *et al.* 1946; Pavlo *et al.* 2003). The structure of wood also changes owing to decomposition of

hemicelluloses, ramification of lignin, and crystallization of cellulose. These changes improve the dimensional stability of wood, increasing its resistance to micro-organisms, darkening its color, and modifying its hardness (Kocaefe *et al.* 2008).

Pinus densiflora and *Larix kaempfi* are the subjects of major tree-planting efforts in Korea (Korea forest service. 2014); *Larix kaempfi* is well-known for decay resistance, while *Pinus densiflora* has poor resistance (Forestry research institute, Korea. 1994). Major domestic research programs for improving wood quality using heat treatment have also been established for the species of *Liriodendron tulipifera* and *Pinus koraiensis* in Korea (Kim *et al.* 2010; Yoon *et al.* 2009; Chang *et al.* 2012; Lim *et al.* 2014). The benefits of low temperature heat treatment for *Pinus densiflora* (red pine) infected by pine-wood nematodes have been reported by Eom *et al.* (2007 and 2008), and an improvement in sound absorption properties has been reported by Byeon *et al.* (2010). However, there has not been much research on improving the mechanical quality of *Pinus densiflora* by using heat treatment. In this study, the effects of heat treatment on the bending strength and compressive strength of *Pinus densiflora* and *Larix kaempferi* were investigated.

2. MATERIALS and METHODS

2.1. Material

Two wood species, red pine (*Pinus densi-*

flora) and larch (*Larix kaempferi*) that have been dried in a natural room temperature condition were used. Samples of 20 × 20 × 320 mm for bending test were cut from each of two species and samples of 20 × 20 × 60 mm for compressive test were also cut from each of two species. The number of samples of the two species was 10 for each heating condition. The humidification of samples was treated over six weeks in a constant temperature and humidity room (20°C ± 1°C, 65% RH ± 2%). The heat treatment of samples was accomplished at the heating rate of 10 °C/min and kept at maximum temperature for 3 h, 6 h, 12 h or 24 h, depending on heating condition in a dry oven (MUFFLE FURNACE, JEIO Tech., Korea). The heat treatment of wood was accomplished at various conditions of 180°C-12 h, 180°C-24 h, 210°C-3 h, 210°C-6 h, respectively. After heat treatment, the samples were humidified again over 2 weeks in a constant temperature and humidity room. And then volume changes, bending properties and compressive strength of the samples were measured.

2.2. Measurement of wood volume change

The volume changes of before and after the heat treatment for each sample were measured by electronic calipers. The percentage of wood volume change (P_v) before and after heat treatment was calculated using the equation (1):

$$P_v = \frac{V_1 - V_0}{V_0} \dots\dots\dots (1)$$

where V_0 and V_1 are the volume before and after heat treatment.

2.3. Measurement of bending and compressive properties

In order to assess the effects of heat treatment parameters on the mechanical properties, bending properties such as modulus of rupture (MOR), static modulus of elasticity (MOE_s), and dynamic modulus of elasticity (MOE_d), and the compressive strengths were measured for the control samples of non-heat treatment, and heat-treated samples under various heating temperatures and times. A static bending test was performed on the samples using a universal testing machine (UTM) (TSU-2, Taeshin Accuracy Machine, Korea) with a centerpoint loading method (concentrated load at mid span and supported at the ends). The size of sample was 20 × 20 × 320 mm and the span was 280 mm and the crosshead speed was set at 2.5 mm/min. Both MOE_s and MOR values were calculated from load-deflection curves of the tests.

A compressive strength test was carried out according to the procedure of a Korean standard (KS F2206, 2004) using a hydraulic testing machine (EHF-ED10-20L, Shimazu, Japan). The size of sample was 20 mm (T) × 20 mm (R) × 60 mm (L). The loading speed was set at 1 mm/min. The load-deformation data obtained were analyzed to determine the compressive modulus of elasticity (MOE_c) and compressive modulus of rupture (MOR_c).

2.4. Measurement of dynamic MOE

In order to measure dynamic MOE (MOE_d), the resonance frequency was first measured by a free transverse vibration at both ends system apparatus which was composed of sine generator (B&K, 1023), universal counter timer (GSP, 5001), and oscilloscope (HP, 1740A). The value at frequency counter timer was measured when the relative amplitude indicated the highest value on the oscilloscope. Resonance frequency (f) MOE_d were calculated by the following equations (2, 3):

$$f = f_0 \left(\frac{1 + \alpha h^2}{l^2} \right) \dots\dots\dots (2)$$

where f = resonance frequency; f_0 = the value obtained from the frequency counter timer; $\alpha = 8.2$, the value according to the chosen vibration type; h = thickness of sample (mm); and l = length of the sample (mm).

And then, MOE_d was calculated by the equation (3):

$$MOE_d = \frac{48\pi^2 \rho l^4 f^2}{m^4 h^2} \dots\dots\dots (3)$$

where ρ = density (Mg/m^3); $m = 4.73$, the value according to the chosen basic vibration.

3. RESULTS and DISCUSSION

3.1. Bending Properties

Table 1 shows the average values of the

bending MOR, static MOE (MOE_s) and dynamic MOE (MOE_d) for two species after heat treatment. As the temperature and time were increased, bending properties of the heat-treated woods after heat treatment decreased. MOR values of red pine wood at heating conditions at 180°C for 12 h and 24 h, and at 210°C for 3 h and 6 h decreased 30.83%, 45.49%, 46.85% and 65.98%, respectively. And MOR values of larch wood at the same condition also decreased 33.43%, 35.10%, 35.74% and 61.75%, respectively.

For MOR in both species, significant differences were observed at 210°C for 3 h and 6 h in the statistical analysis.

MOE_s and MOE_d of two species of wood after heat treatment were also dropped with an increase in the heating temperature and time. These results indicated that the reduction of MOR was higher than those of MOE_d and MOE_s .

And the changes of bending properties showed almost same trend in both species. However, the effects of heat treatment on bending properties for red pine were greater than those for larch because the reduction values for red pine were higher than those for larch with the exception of MOE_s at 180°C.

The effect of heat treatment at a high temperature on the bending MOR was greater in both species than that for a long time, because the MOR value was the lowest at 210°C, 6 h and the MOR value at 180°C, 24 h was similar to that at 210°C and 3 h.

Kocaefe *et al.* (2010) reported that there was

Table 1. Bending strength performance of heated-treated wood

Wood species	Heat treatment conditions	N	MOR			MOE _d			MOE _s		
			Mpa	<i>p</i> value	P _{MOR} ²⁾ (%)	GPa	<i>p</i> value	P _{MOE_d} ³⁾ (%)	GPa	<i>p</i> value	P _{MOE_s} ⁴⁾ (%)
<i>Pinus densiflora</i>	Control	10	247.23 a ¹⁾ (19.18)	-	-	11.17 a (0.69)	-	-	11.01 a (0.74)	-	-
	180°C, 12 h	10	171.01 b (14.50)	0.013	-30.83	10.29 b (0.20)	0.003	-7.88	10.40 ab (0.28)	0.212	-5.54
	180°C, 24 h	10	134.77 c (24.28)		-45.49	10.52 ab (0.44)		-5.85	10.03 b (0.62)		-9.42
	210°C, 3 h	10	131.40 c (16.51)	0.000	-46.85	9.99 b (0.73)	0.010	-10.56	9.51 b (1.08)	0.009	-14.96
	210°C, 6 h	10	84.11 d (12.03)		-65.98	8.71 c (0.68)		-22.02	7.70 c (0.84)		-34.81
<i>Larix kaempferi</i>	Control	10	262.02 a (17.77)	-	-	13.07 (0.51)	-	-	12.43 a (0.30)	-	-
	180°C, 12 h	10	174.43 b (28.35)	0.880	-33.43	12.57 (0.70)	0.009	-3.83	10.97 b (0.94)	0.312	-11.75
	180°C, 24 h	10	170.04 b (56.41)		-35.10	12.81 (0.23)		-1.99	10.45 b (0.50)		-15.93
	210°C, 3 h	10	168.37 b (26.70)	0.003	-35.74	13.04 (0.81)	0.108	-0.23	10.60 b (0.54)	0.059	-14.72
	210°C, 6 h	10	100.21 c (29.03)		-61.75	11.70 (1.50)		-10.48	9.12 c (1.44)		-26.63

Notes: ¹⁾ means with the same letter are not significantly different at a *p* value of 0.05 according to Duncan's new multiple range test, parenthesis is standard deviation,

MOR: Modulus of rupture, MOE_d: Dynamic modulus of elasticity, MOE_s: Static modulus of elasticity,

P_{MOR}²⁾: Percentage in MOR value after heat treatment,

P_{MOE_d}³⁾: Percentage in MOE_d after heat treatment,

P_{MOE_s}⁴⁾: Percentage in MOE_s after heat treatment.

a significant change in mechanical properties (MOE, MOR, hardness, etc.) due to heat treatment. This deterioration might be explained as due to hemicellulose degradation. Yildiz *et al.* (2006) reported that the heat treatment and control samples were tested for some mechanical properties; compression strength, bending strength, modulus of elasticity in bending, hardness, impact bending strength and tension strength perpendicular to grain. The results indicated that heat treatment samples had lower mechanical properties compared to the

control samples. It was seen that hemicelluloses were the wood-cell components most degraded by the heat treatment. Also, Bekir (2014) reported that black pine wood showed compared to the control samples, MOR and impact bending strength values decreased as the heat treatment temperature increased. And the dynamic modulus of elasticity decreased by about 13% in the most severe treatment at 230°C for 4 h (Garcia *et al.* 2012).

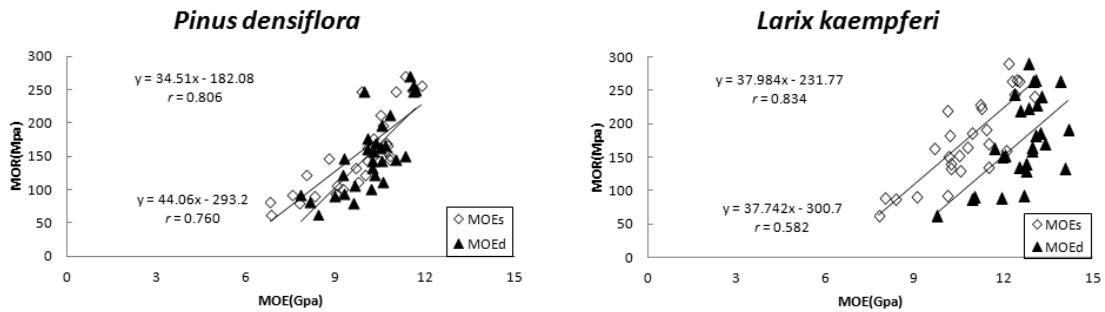


Fig. 1. Relation of bending MOE to MOR of heat-treated wood.

3.2. The relations of bending MOR to MOE_d and MOE_s of heat-treated wood

Fig. 1 shows relations of between MOE_d and MOR, and MOE_s and MOR for red pine and larch woods after heat treatment. Correlation coefficients of MOR to MOE_s and MOE_d for red pine woods were 0.806 and 0.760, respectively. And Correlation coefficients of MOR to MOE_s and MOE_d for larch woods were 0.834 and 0.582, respectively. Regression coefficients of both species of wood were significant at a p value of 0.01 level, and similar regressions were also found between MOR and MOE_d, and MOR and MOE_s. Won *et al.* (2013) reported that there were a high correlation at 1% level between MOR and MOE_d, and MOR and MOE_s of heat-treated wood. And Nakai (1984) also reported that solid Japanese cedar showed a high correlation coefficient (0.69-0.78) between MOE and MOR. Thus, high relationships found in this study are quite reasonable when compared to those published in literature (Nakai 1984; Won *et al.* 2013).

3.3. Compressive properties of heat-treated wood

Table 2 shows the average values of the volume, compressive strength and compressive MOE_c for two species of wood after heat treatment. The volume and bending strength of both species of wood after heat treatment decreased with an increase in the heating temperature and time. However, the compressive strengths of both species of wood that had been heat-treated at 180°C for 12 h and 24 h, and at 210°C for 3 h and 6 h were greater than those of the control samples. For volume in both species, significant differences were observed at 210°C for 3 h and 6 in the statistical analysis.

Michiel *et al.* (2007) reported that, by heat treatment, longitudinal compressive strength for softwoods increased clearly. The increase of the compressive strength in longitudinal direction might be due to a lower amount of bound water in heat-treated wood.

Table 2. Compressive properties of heat-treated wood

Wood species	Heat treatment conditions	N	Volume			MOR		MOE	
			cm ³	<i>p</i> value	P _V ¹⁾ (%)	MPa	<i>p</i> value	GPa	<i>p</i> value
<i>Pinus densiflora</i>	Control	10	24.43 a (0.07)	-	-	44.48 (4.05)	-	4.32 (0.54)	-
	180 °C, 12 h	10	23.56 b (0.18)	0.151	-2.81	48.99 (5.60)	0.741	4.17 (0.63)	0.856
	180 °C, 24 h	10	23.44 b (0.34)		-4.17	49.89 (3.97)		4.22 (0.49)	
	210 °C, 3 h	10	23.52 b (0.14)	0.027	-3.25	51.01 (3.84)	0.550	4.67 (0.40)	0.076
	210 °C, 6 h	10	23.29 c (0.22)		-4.35	49.32 (6.79)		4.13 (0.70)	
<i>Larix kaempferi</i>	Control	10	24.35 a (0.08)	-	-	44.42 (6.04)	-	3.97 (0.90)	-
	180 °C, 12 h	10	23.41 b (0.38)	0.656	-2.90	60.89 (6.98)	0.184	4.96 (1.22)	0.142
	180 °C, 24 h	10	23.57 b (0.49)		-3.08	51.57 (3.46)		3.51 (1.32)	
	210 °C, 3 h	10	23.46 b (0.26)	0.003	-2.94	56.57 (6.76)	0.731	4.41 (1.34)	0.506
	210 °C, 6 h	10	22.81 c (0.40)		-6.02	56.79 (7.00)		5.06 (0.38)	

Note; P_V¹⁾: Percentage in volume after heat treatment.

4. CONCLUSION

This study was conducted to assess the effects of heat treatment at various temperatures and times, on the volume change, bending and compressive performance of two species of wood. The results showed that there were significant changes in bending properties such as modulus of elasticity (MOE_d MOE_s), modulus of rupture (MOR) and compressive strength after heat treatment. The volume and bending strength of both species of wood after heat treatment decreased with an increase in the heating temperature and time. The effect of heat treatment at a high temperature on the bending MOR was greater in both species than

that for a long time. However, the compressive strengths of two species of wood that had been heat-treated at 180 °C for 12 h and 24 h, and at 210 °C for 3 h and 6 h were greater than those of the control samples. Furthermore, highly significant correlations were observed between MOR and MOE_d and MOR and MOE_s for all heating conditions.

ACKNOWLEDGEMENT

This study was carried out with the support of 'Research center for ecological restoration in damaged land (Project No. S211214L020410)' provided by Korea Forest Service.

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