Effects of Heat Treatment on the Mechanical Properties of Loblolly Pine, Sweetgum, and Red Oak

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Abstract

Heat treatment has been assessed as an eco-friendly means to reduce wood's dimensional instability and biodeterioration under varying ambient conditions. However, irreversible losses in mechanical properties have been reported to occur when wood is subjected to elevated temperatures as a result of the autocatalytic breakdown of the cell-wall constituents. The reduced strength of heat-treated wood is a disincentive for its extensive use in structural applications where high-strength quality is important. Thus, improved durability must be balanced with the preservation of the mechanical properties of heattreated wood. The strength properties of heat-treated loblolly pine (Pinus taeda), sweetgum (Liquidambar styraciflua), and water oak (Quercus nigra) were assessed to quantify the effects of varying temperature levels, up to 204°C, and duration, up to 8 hours, on flexural strengths, compression strengths, shear strength, and hardness. Two-factor analysis of variance (ANOVA) and 1-factor ANOVA (where there is temperature–duration interaction) of results coupled with Tukey's pairwise comparison (α = 0.05) indicated effects ranging from indicative differences in modulus of elasticity to significant increment and decrement in other strength parameters. Generally, strength properties for the three species were optimally preserved, or peaked and dominantly improved in specimens treated at 149^oC for 8 hours. This study provides a knowledge base for process optimization in heat treatment industries and for the use of heat-treated wood in structural applications.

I he versatility of wood and wood products across multiple applications has made it indispensable despite the rapid evolution of substitute or alternative materials like plastic, iron, and steel. Wood, however, has a biodegradable nature and is susceptible to dimensional changes while in service, especially in exterior applications where changing moisture conditions are inevitable. The cellulose content of wood serves as a substrate for the growth of fungi and mold, which deteriorates both the physical and mechanical properties of wooden structures.

Technological innovations have improved the utility of wood through re-engineering in order to derive more products that satisfy consumer's often diverse aesthetic and functional tastes. Also, the negative influences of moisture and biological agents have been investigated and considerably reduced through many previous studies (Chidester 1937, Dwinell and Carr 1995, Militz and Tjeerdsma 2001). These include, among others, chemical and pressure treatment of wood, painting of logs, drying of wood, and heat treatment of wood. Heat treatment may be the oldest method: in ancient times, edges of fence poles were heated to increase durability (Jamsa and Viitaniemi 2001), and recently there have been continuing attempts to improve wood by thermal treatment, especially in Finland, France, and some other European countries (Dirol and Guyonnet 1993, Troya and DeNavarrete 1994, Viitanen et al. 1994, Boonstra et al. 1998, Tjeerdsma et al. 1998).

Industrial-scale heat treatment of wood was developed at the Technical Research Centre of Finland in the early 1990s, and the total production capacity of heat-treated wood in 2002 was estimated to be approximately $265,000 \text{ m}^3$ (Korkut et al. 2008b). Efforts to improve thermal treatment of wood have led to the development of several treatment processes, and materials produced through thermal treatments have been introduced to the European market. This has resulted in the development of Thermowood (Stellac) in Finland (Viitanen et al. 1994), Torrefaction (Perdure) in France (Weiland and Guyonnet, 1997), and Plato-wood in The Netherlands (Syrjanen and Oy 2001).

Heat treatment improves the dimensional stability and biodurability of wood. Mold and fungi require suitable

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temperature, nutrients, oxygen, and moisture (Robbins and Morrel 2006) to infest and thrive on materials that serve as the food substrate source. Thermal treatment reduces the hygroscopic properties of wood (Weiland and Guyonnet 2003) by facilitating the degradation of hemicelluloses (Boonstra and Tjeerdsma 2006). Moreover, the overall holocellulose content becomes modified and caramelized under high temperature (Boonstra and Tjeerdsma 2006), thus rendering it unsuitable as a food substrate for insects and molds. Consequently, the resistance of wood to biological attacks is greatly enhanced (Momohara et al. 2003, Kocaefe et al. 2007, Tubajika et al. 2007).

Loblolly pine (Pinus taeda), water oak, a member of the red oak group (Quercus nigra), and sweetgum (Liquidambar styraciflua) are tree species of different qualities that are representative of species found in Arkansas and much of the United States. We selected these three species because they capture the wide variation that exists in the cellular structure of wood species, especially in United States. This ranges from the softwood structure dominated by tracheids, as in Loblolly pine; to the ring-porous hardwood composition of vessel and fiber tracheid, as in the red oak group; and to the diffused-porous hardwood composition, as in sweetgum. Heat treatment of these wood species will in no small measure improve their durability and stability for both indoor and outdoor applications. However, since the impact of the heat treatment process also depends on the type of wood species (Boonstra et al. 2006), there is a need to determine the degree of tolerance of their individual mechanical strength properties to elevated treatment temperatures. This will facilitate a good understanding of optimum application level and duration of heat treatment to these species for industrial and commercial purposes.

This study was conducted to assess the effect of different levels and duration of heat treatment on the mechanical properties of selected wood species and to recommend heat treatment levels at which optimum mechanical strength is preserved for the selected species.

Materials and Methods

A total of three healthy trees were selected (one for each species), felled at 15-cm stump, and cut into four 2.44-m sections (bolts) by length. The bolts were appropriately marked and skidded to the sawmill, where they were quarter-sawn to achieve the proper growth ring orientation on a Woodmizer sawmill into 6.35-cm (thickness) flitches with random width and 2.44-m length.

The flitches were conveyed into a dehumidification dry kiln, which was operated so that kiln-drying defects such as case hardening, honey-combing, or collapse was avoided. The initial setting was 32° C and 40 percent condenser time. After the surface moisture was removed and the relative humidity dropped, the settings were 38° C and 50 percent condenser time. When the temperature and relative humidity indicated an equilibrium moisture content (EMC) below fiber saturation point, the settings were changed to 49° C and 60 percent. When the indicated EMC was 10 percent, the kiln was secured and the circulating fans were left on for 3 days to level the moisture gradient in the wood at an EMC of 12 percent.

The flitches were planed to 5.10-cm thickness, ripped, planed to 5.10-cm width, and cut into different lengths with respect to the sample dimensions for each test according to ASTM D143 specifications (76.2 cm for static bending, 20.3 cm for compression parallel to the grain, 15.2 cm for compression perpendicular to the grain and hardness, and 6.4 cm for shear; ASTM International 2004). The ripping operation started from the outside edge of the flitch and moved inward so as to use mature wood and eliminate juvenile wood.

The initial treatment conditions of our control samples are similar to conventional kiln drying conditions and are designated as a reference point for further treatments applied. This is because the heat-treated wood would be competing in the marketplace against commercially kilndried wood. Thus, the control specimens are considered "untreated" since all commercially available wood has a similar temperature history. Southern pine is often kiln dried at 116°C, while the hardwoods are often dried at a temperature of 82 $^{\circ}$ C; therefore, 93 $^{\circ}$ C, an average of the three kiln dry temperatures, was selected as the control temperature. The control duration was arbitrarily chosen at 2 hours, since at higher temperatures it would be important to see how quickly the wood reacts to the higher temperatures. The control samples used in this study were those of the control temperature $(93^{\circ}C)$ and control duration (2 h).

The specimens for each species were sorted out randomly into nine treatment groups constituted by a combination of three temperature levels (93 $^{\circ}$ C, 149 $^{\circ}$ C, 204 $^{\circ}$ C) and three duration times (2, 5, 8 h) for each strength property assessed. Ten replicates per treatment combination were designated for each strength test per species, with a total of 270 samples for each strength property tested except for static bending, which totaled 252 samples (eight replicates per treatment combination were used for Sweetgum static bending to maintain source-homogeneity of the samples despite the slight insufficiency of the wood material). Properties vary with tree height but differently by species. Care was taken to ensure that the random selection per treatment combination would yield 10 replicates drawn from all positions within the tree. The mean of the 10 replicates should approximate the tree average and thus remove tree position as a variable in the analysis.

The heat treatment process was conducted in the Wisconsin Corp. Automatic Thermo-regulated Industrial Oven, where the treatment temperature level was digitally programmed as the set-point (SP) of the oven and allowed to run for 24 hours in order to attain and stabilize the process value at the SP with a zero deviation on the display screen. The wood specimens belonging to the treatment batch of the set-point temperature were afterward manually conveyed into the oven. At the expiration of each treatment duration (i.e., 2, 5, and 8 h), the batch that belonged to each treatment duration was removed from the oven and allowed to cool down. After all heat treating was completed, the specimens were conveyed to the conditioning chamber, where they were conditioned to attain an EMC. The chamber was set at a temperature of $21^{\circ}C (\pm 1^{\circ}C)$ and a relative humidity of 65 percent $(\pm 3\%)$ at which untreated wood would attain an EMC of 12 percent. The shortest specimens (shear) were removed for testing from the chamber after 4 weeks. Testing sequence continued through the specimens by increasing length. The longest specimens (static bending) were in the chamber for 12 weeks.

Six different strength properties that are crucial for load bearing structures were tested. The different strength tests were conducted in compliance with the standards for testing small wood samples (ASTM D143-94; ASTM International

2004). The tests and corresponding ASTM D143 sections are modulus of rupture (MOR), section 8; modulus of elasticity (MOE), section 8; compression perpendicular to grain, section 12; compression parallel to grain, section 9; shear strength, section 14; and hardness, section 13. It should be noted that the MOE and MOR calculations were based on load-deflection curves from the static bending procedure. Test specimen sizes were stated earlier. All tests were carried out using different ancillary equipment coupled to the Instron Universal Testing Machine model 4400, while the load was applied at a stipulated uniform rate of crosshead beam movement. Before mounting each specimen for testing, the actual cross-section dimension and length was measured with Mitutoyo electronic vernier calipers. Wood samples were weighed before the test, dried in the oven at 103° C for 24 hours after the test, and reweighed in order to estimate the test moisture content of each sample.

Results obtained from each strength test were initially analyzed using a 2-factor analysis of variance (ANOVA) in order to determine whether the effects of temperature and duration on each strength property were separable while evaluating the significance of differences. There were significant interactions ($\alpha = 0.05$) between temperature and duration effects on some strength properties, which implies that they jointly impact such strength properties and their effects are inseparable. Thus, each temperature was paired with different durations to obtain nine different treatment levels, and 1-way ANOVA was used to determine significant differences in the effect of the treatment combinations on the strength properties. Tukey's test was used to estimate the significance of pairwise differences of mean values.

Results and Discussion

Modulus of rupture

The effects of temperature (θ) and duration (λ) on the MOR of the three wood species are inseparable because of the significance of the interaction ($P(\theta \times \lambda) < 0.0001$ for pine and sweetgum, and $P(\theta \times \lambda) = 0.0002$ for oak); hence, the results were analyzed using 1-way ANOVA. The results obtained show that there are significant differences in the effects of the treatment combinations on the MOR across the three species. Generally, highest average MOR was observed on samples treated at 149° C for 8 hours (Table 1), while the MOR was significantly reduced for samples treated at 204 °C for 8 hours. The MOR for oak improved

steadily until the peak value was attained, while the MOR for pine and sweetgum fluctuated (Fig. 1). The graphs for oak and sweetgum suggest that MOR generally improves as the specimens are subjected to heat treatment conditions up to the limit of 149° C for 8 hours. Pine is less indicative of this.

Modulus of elasticity

The effects of temperature (θ) and duration (λ) on MOE are separable for the three species as indicated by the insignificance of their interaction term under the 2-factor ANOVA (P (θ \times λ) = 0.2841 for pine, 0.9481 for sweetgum, and 0.4382 for oak). Temperature and duration did not have significantly different effects on the MOE of pine and oak, but they did have significantly different effects on the MOE of sweetgum (Table 2). The highest average MOE was recorded in sweetgum and oak specimens treated at 204°C for 5 hours, while pine specimens attained peak MOE at 149° C for 8 hours (Fig. 2). High treatment temperature of 204° C for 8 hours did not significantly compromise the original MOE of the three species, and the observed intermediate increments were indicative but negligible variations.

Figure 1.—Modulus of rupture of heat-treated pine, sweetgum, and oak. Vertical dashed lines represent quasi-Arrhenius partitions between temperature groups; error bars indicate standard errors.

Table 1.—Tukey pairwise comparison results of modulus of rupture (MOR) for heat treated pine, sweetgum, and oak.

			MOR (MPa) ^a						
Temperature $(^{\circ}C)$	Duration (h)		Pine		Sweetgum		Oak		
		Mean	CV	Mean	CV	Mean	CV		
93	2	94.32 BA	10.10	95.19 BC	6.26	111.56 DC	3.55		
	5	87.43 BA	14.29	99.31 BA	3.44	117.77 BDC	4.26		
	8	93.23 BA	12.39	101.11 BA	3.07	119.25 BDC	4.52		
149	$\overline{2}$	102.71 A	11.34	94.75 BC	8.54	128.27 BAC	6.42		
	5	99.39 A	7.26	103.12 BA	8.70	139.40 A	10.06		
	8	102.97 A	8.08	110.66A	7.44	140.53 A	5.95		
204	2	95.52 BA	12.98	105.99 A	6.51	133.78 BA	10.58		
	5	79.15 B	29.10	98.63 BA	13.59	134.39 BA	13.54		
	8	57.43 C	26	81.51 C	16.23	107.60 D	26.41		

 $a_n = 10$ for pine and oak; $n = 8$ for sweetgum. Homogenous groups: within a species, means followed by different letters are significantly different according to the Tukey test at $P < 0.05$. CV = coefficient of variation.

Table 2.—Modulus of elasticity (MOE) of heat-treated pine, sweetgum, and oak.

	MOE (GPa) ^a					
	Pine		Sweetgum		Oak	
	Mean	CV	Mean	CV	Mean	CV
Temperature $(^{\circ}C)$						
93	13.30 A	11.74	13.67 B	7.75	20.67A	8.77
149	14.28 A	9.81	14.14 BA	8.20	21.16 A	12.08
204	13.32 A	16.80	14.82 A	10.65	21.98 A	7.82
\boldsymbol{P}	0.2119		0.0047		0.1515	
Duration (h)						
\overline{c}	13.80 A	12.22	13.81 B	7.73	21.40 A	8.04
5	13.67 A	11.98	14.71 A	9.54	21.27 A	13.26
8	13.42 A	15.73	14.11 BA	10.29	21.14 A	7.79
\overline{P}	0.7650		0.0110		0.8899	

 $a_n = 30$ for pine and oak; $n = 24$ for sweetgum. Homogenous groups: within a species, means followed by different letters are significantly different according to the Tukey test at $P < 0.05$. CV = coefficient of variation.

The measured MOEs are in agreement with previous studies that show varying increase in stiffness. MOEs of different species have been reported to be less affected by heat treatment (Kamdem et al. 2002, Esteves et al. 2007, Mburu et al. 2008, Esteves and Pereira 2009), increased slightly at treatment temperatures of 180° C to 200° C (Inoue et al. 1993), and increased greatly as in Eucalyptus globulus (Santos 2000). The statistical insignificance of the difference between the MOEs of specimens under control treatment conditions and peak treatment conditions across the three species suggests that MOE is greatly preserved under dry and relatively mild heat treatment.

Compression perpendicular to grain

The effects of temperature (θ) and duration (λ) on the resistance to perpendicular compression of the three wood species are inseparable as a result of the significance of the interaction (P ($\theta \times \lambda$) = 0.0181 for pine, 0.0016 for sweetgum, and 0.0022 for oak). Perpendicular compression strength within each species was significantly different ($P =$

Figure 2.—Modulus of elasticity of heat-treated pine, sweetgum, and oak. Vertical dashed lines represent quasi-Arrhenius partitions between temperature groups; error bars indicate standard errors.

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0.0012 for pine, $P = 0.0125$ for sweetgum, and $P = 0.0002$ for oak). Highest values were observed at treatment temperatures of 149° C for 8 hours duration for pine, 93° C for 8 hours for sweetgum, and 204° C for 2 hours for oak. In five of nine cases, there is no significant pairwise difference between all treatment levels except 204°C-2 hours and 204° C -8 hours in pine, 204° C -8 hours in sweetgum, and 93° C -2 hours and 204° C -5 hours in oak (Table 3). Even though there were irregular increments at the different temperature and duration combinations (Fig. 3), there was an overall decrease from control $(93^{\circ}C-2)$ h) to maximum $(204^{\circ}C-8)$ h) treatment of 7.94 percent for pine and a decrease of 1.47 percent for sweetgum, while oak had an overall increase of 12.64 percent. The decreases might be attributable to the decrease in density, as observed in similar species of Scots pine (*Pinus sylvestris*; 10%) and Norway spruce (Picea abies; 8.5%; Boonstra et al. 2007). The increase in the perpendicular compression of oak could be explained by the joint effect of lower equilibrium moisture content based on the lower sorption properties of hardwoods, the formation of new chemical bonds within the cell wall due to reticulation of the lignin matrix, and reported crystallization of cellulose at relatively low temperature range (Bhuiyan et al. 2000).

Compression parallel to grain

In most cases heat treatment had a positive effect on resistance to compression parallel to the grain (Fig. 4). Temperature (θ) and duration (λ) were separable for pine and oak $(P (\theta \times \lambda)) = 0.1427$ for pine and 0.4153 for oak) but inseparable for sweetgum (P ($\theta \times \lambda$) = 0.0003). Thus, pine and oak results were analyzed using 2-factor ANOVA, while sweetgum results were analyzed with 1-way ANOVA. Pairwise comparison of parallel compression values showed that the different temperature and duration levels have no significantly different effects on the parallel compression strength of pine. The parallel compression of oak was significantly different only at 93° C, but not at any of the duration times (Table 4). The compression strength in the longitudinal direction of sweetgum was significantly different among the nine treatment combinations ($P <$ 0.0001), while the pairwise comparison shows that values obtained at 204° C-8 hours and 93° C-2 hours are significantly different from the maximum average strength

Table 3.—Tukey pairwise comparison result of compression perpendicular to the grain for heat-treated pine, sweetgum, and oak.

	Duration (h)		Perpendicular compression (MPa) ^a						
			Pine		Sweetgum		Oak		
Temperature $(^{\circ}C)$		Mean	CV	Mean	CV	Mean	CV		
93	$\overline{2}$	12.20 BAC	14.64	10.13 BA	10.45	12.83 C	10.84		
	5	12.34 BAC	23.99	11.06 BA	14.66	13.92 BAC	9.35		
	8	13.88 BA	25.69	12.52 A	19.61	14.36 BAC	6.78		
149	Ω	11.86 BAC	41.07	12.14 BA	12.26	14.60 BAC	6.75		
	5	13.20 BAC	23.51	11.27 BA	10.63	15.74 BA	12.26		
	8	16.02 A	17.74	11.58 BA	11.27	15.87 A	17.24		
204	\overline{c}	11.23 BC	22.19	12.25 BA	13.35	15.99A	8.00		
	5	11.87 BAC	30.32	11.26 BA	22.86	13.37 BC	17.21		
	8	9.12 C	22.46	9.98 B	15.90	14.45 BAC	8.79		

 $a_n = 10$ for all cases. Homogenous groups: within a species, means followed by different letters are significantly different according to the Tukey test at $P <$ 0.05. $CV = coefficient of variation.$

Figure 3.—Compression perpendicular to grain of heat-treated pine, sweetgum, and oak. Vertical dashed lines represent quasi-Arrhenius partitions between temperature groups; error bars indicate standard errors.

Figure 4.—Compression parallel to grain of heat-treated pine, sweetgum, and oak. Vertical dashed lines represent quasi-Arrhenius partitions between temperature groups; error bars indicate standard errors.

 $a_n = 30$ for all cases. Homogenous groups: within a species, means followed by different letters are significantly different according to the Tukey test at $P < 0.05$. CV = coefficient of variation.

 $a_n = 10$ for all cases. Homogenous groups: in pairwise comparisons, means followed by different letters are significantly different ($P < 0.05$). CV = coefficient of variation.

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Table 6.—Pairwise comparison of the shear strength of heattreated pine.

	Shear strength (MPa) ^a	
	Mean	CV
Temperature $(^{\circ}C)$		
93	9.62 A	16.55
149	9.11 A	23.11
204	5.57 B	35.79
\overline{P}	0.0082	
Duration (h)		
$\overline{2}$	8.74 A	21.07
5	8.34 A	28.44
8	7.22A	45.27
\boldsymbol{P}	0.1904	

 $a_n = 30$ for all cases. Homogenous groups: means followed by different letters are significantly different according to the Tukey test at $P < 0.05$. $CV = coefficient of variation.$

93C-2h 93C-5h 93C-8h 149C-2h 149C-5h 149C-8h 204C-2h 204C-5h 204C-8h **Temperature and Duration Combination**

Temperature and Duration Combination

Figure 6.—Janka hardness of heat-treated pine (A), sweetgum (B), and oak (C). Vertical dashed lines represent quasi-Arrhenius partitions between temperature groups; error bars indicate standard errors.

 $a_n = 10$ for all cases. Homogenous groups: within a species, means followed by different letters are significantly different according to the Tukey test at $P < 0.05$. CV = coefficient of variation.

Figure 5.—Shear parallel to grain of heat-treated pine, sweetgum, and oak. Vertical dashed lines represent quasi-Arrhenius partitions between temperature groups; error bars indicate standard errors.

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obtained (Table 5). These results are closely related to the results obtained for perpendicular compression. Hence, it further lends credence to the inference that heat treating at 204° C for 8 hours does not have a significant negative influence on the compression strength of the wood species.

Shear strength

Temperature (θ) and duration (λ) effects on shear strength are inseparable for sweetgum and oak ($P(\theta \times \lambda) < 0.0001$), while they are separable for pine $(P (\theta \times \lambda) = 0.0624)$. In pine, shear strength was not significantly different at all durations, but it was significantly different at the temperature level of 204° C (Table 6). The pairwise comparison shows that the effect of heat treatment on the shear strength of sweetgum was not significantly different within the range of 93 \degree C-2 hours to 204 \degree C-2 hours, but was different at 204° C -5 hours and 204° C -8 hours. Similar results were obtained for oak, but the range was between 93° C-2 hours to 149° C–8 hours, while the shear strength at 204° C–2 hours compares with some of the treatment levels within the range (Table 7). Maximum average shear strength was observed at treatment levels of 149° C for 2 hours for both sweetgum and oak, while it was observed at 93° C and a duration of 2 hours for pine. There seems to be a similar rate of decline across the three species, which suggests a nondifferential influence of high temperature on the shear strength irrespective of the wood species (Fig. 5).

There was an immense decline in shear strength of 60 percent in pine, 72 percent in sweetgum, and 55 percent in oak at the peak treatment condition as compared with the shear strength at control conditions. These results compare with significant differences observed in tangential and radial shear strength of European aspen (Populus tremula) and hybrid aspen (Herajarvi 2009); however, the depreciation percentages were smaller (10% to 15%) presumably because of the difference in heat treatment process adopted.

Janka hardness

Temperature and duration effects were separable for the tangential, radial, and longitudinal hardness of pine and for the longitudinal hardness of oak; however, they were inseparable for all directions of sweetgum and for the radial and tangential directions of oak.

Pine showed an indicative increase in hardness up to 149 $^{\circ}$ C in the three directions, while at 204 $^{\circ}$ C there was a significant decrease in the tangential hardness, insignificant increase in the radial hardness, and significant increase in the longitudinal hardness (Fig. 6A). Highest but insignificant duration effects were observed at 5 hours in tangential and radial hardness and at 8 hours for longitudinal hardness

Table 8.—Janka hardness values of heat-treated pine based on temperature and duration.

	Janka hardness (kN)					
	Tangential		Radial		Longitudinal	
	Mean	CV	Mean	CV	Mean	CV
Temperature $(^{\circ}C)$						
93	3.33A	26.10	3.39A	23.87	4.57 B	18.03
149	3.37 A	25.95	3.36A	32.54	4.65 B	17.34
204	2.70 B	23.07	3.53 A	29.42	5.49 A	19.78
\boldsymbol{P}	0.0095		0.4858		0.0172	
Duration (h)						
\overline{c}	$3.30\,\mathrm{A}$	23.13	3.35A	26.86	4.75 A	20.77
	2.99A	26.52	3.48 A	22.54	4.84 A	18.60
8	3.09 A	31.22	3.45 A	35.63	5.12 A	21.27
\overline{P}	0.1478		0.6877		0.2659	

 $a_n = 30$ for all cases. Homogenous groups: means followed by different letters are significantly different ($P < 0.05$). CV = coefficient of variation.

 $a_n = 10$ for all cases. Homogenous groups: in pairwise comparisons, means followed by different letters are significantly different ($P < 0.05$). CV = coefficient of variation.

(Table 8). This is somewhat similar to the findings of Boonstra et al. (2007), who reported a significant increase of 48 percent in Brinell hardness of heat-treated Scots pine. In contrast to the sustained reduction in the Janka hardness of Scots pine reported by Korkut et al. (2008a), the radial and longitudinal Janka hardness of pine increased while the tangential hardness decreased at the peak temperature and duration (Fig. 6A). The reduction observed by Korkut et al. (2008a) might be strongly attributable to their obliteration of

Table 10.—Longitudinal Janka hardness of heat-treated oak.

		Janka hardness (kN) ^a				
		Tangential		Radial		
Temperature $(^{\circ}C)$	Duration (h)	Mean	CV	Mean	CV	
93	2	6.59 BA	4.67	6.33 BA	8.74	
	5	6.91 A	9.04	6.64 A	7.42	
	8	6.85 A	5.45	6.28 BA	11.09	
149	\overline{c}	6.97 A	6.68	6.70A	8.74	
	5	6.74 BA	6.92	6.63A	8.74	
	8	7.02A	9.37	6.97 A	8.72	
204	\overline{c}	6.81 A	8.89	6.84 A	8.27	
	5	5.66 C	8.79	5.10 C	9.72	
	8	6.03 BC	6.84	5.58 BC	8.02	

 $a_n = 10$ for all cases. Homogenous groups: in pairwise comparisons, means followed by different letters are significantly different ($P < 0.05$). CV = coefficient of variation.

Table 11.—Longitudinal Janka hardness of heat-treated oak.

	Janka hardness (kN) ^a		
	Mean	CV	
Temperature $(^{\circ}C)$			
93	7.97 A	6.42	
149	7.99 A	7.40	
204	8.34 A	6.64	
\overline{P}	0.2655		
Duration (h)			
\overline{c}	8.13 A	7.30	
5	8.05 A	6.68	
8	8.11 A	7.39	
\overline{P}	0.9265		

 $a_n = 30$ for all cases. Homogenous groups: in pairwise comparisons, means followed by different letters are different ($P < 0.05$). CV = coefficient of variation.

the normal treatment effects on the hardness of the heattreated specimen. They transformed the strength values obtained to a moisture content of 12 percent, which is often unattainable in thermally modified wood as a result of the reduction of its hygroscopic properties.

The highest value for hardness in all directions for sweetgum was observed at 149°C for 8 hours. The observed differences are, however, insignificantly different in comparison with the values obtained from the control samples at 93° C for 2 hours (Table 9). There was an almost identical declination trend in the hardness values after the peak hardness value had been attained (Fig. 6B). It can be deduced from the pairwise comparison results that the original tangential and longitudinal hardness of sweetgum are not significantly affected by heat treatment up to a temperature of 204° C within 5 hours, while the radial hardness is sustained at a limit of 204°C within 2 hours.

The highest values for radial and tangential hardness of oak were observed at 149°C for 8 hours, but the values were not significantly different from the hardness of the control samples (Table 10). Maximum but insignificantly different longitudinal hardness was recorded at 204°C and 2 hours (Table 11). The alternating pattern of increments and decrements as reflected in the tangential, radial, and longitudinal hardness of oak suggests that there is an ongoing transitory modification of the hardness with respect to change in the treatment conditions (Fig. 6C).

The result obtained is comparable to the findings of Kocaefe et al. (2008b), who reported that aspen and birch, which share similar structural characteristics with sweetgum, attained a peak tangential hardness value at 160° C. Also, the observed increments are consistent with previous findings where hardness has been reported to be slightly increased in heat-treated birch (Ponscak et al. 2006).

Moisture content

Generally, it was observed that the specimens subjected to peak treatment conditions attained lower moisture content compared with the specimens under the control treatments even though they were all conditioned to attain an EMC of 12 percent. The reduction in moisture content at peak condition compared with the control treatments was 45 percent in pine, 51 percent in sweetgum, and 56 percent in oak. This is consistent with the conclusions of previous studies (Chirkova et al. 2005, Kocaefe et al. 2008a), where it has been reported that the moisture repellency of wood is greatly enhanced by thermal treatment.

Table 12.—Equilibrium moisture content attained in each species after heat treatment and conditioning in a chamber set for 21°C and 65 percent relative humidity.^a

			Avg. moisture content (%) at different treatment levels	
Temperature $(^{\circ}C)$	Duration (h)	Pine	Sweetgum	Oak
93		10.79	10.80	9.09
		10.29	10.32	8.43
	8	10.04	9.83	7.78
149		9.30	9.38	6.76
		8.91	8.70	6.21
	8	8.46	8.41	5.87
204		7.93	7.81	5.35
		6.61	5.77	4.10
	8	5.96	5.26	3.96

^a $n = 50$ for pine and oak cases; $n = 48$ for sweetgum cases.

It should be noted, however, that the strength values were not adjusted for the changes in EMC at the different treatment levels. This is because any MC-based adjustment will unjustifiably obliterate the regular effect of thermal treatment on the specimens' strength, which is influenced by the irreversible treatment-induced change in attainable EMC. Table 12 shows the final moisture content attained in each species at different treatment levels after being conditioned in a chamber set for normal wood to attain an EMC of 12 percent.

Summary and Conclusions

The results obtained are complementary to previous findings, where it has been reported that the essential strength properties of wood species treated above 160° C decreased significantly (Stamm et al. 1946, Kamdem et al. 2002). However, there were significant increases in MOR up to 149°C for 8 hours, variant significant increases in MOE, intermediate increases in compression strengths, sustained shear strength up to 149° C for 8 hours, and insignificantly improved or sustained hardness values. According to the literature, the crystallinity of cellulose, which is strongly correlated with the strength, is not changed (Yildiz and Gümüskaya 2007) or can even improve up to a certain temperature, which may be as high as 200° C depending on the conditions involved (Bhuiyan et al. 2000, Akyildiz et al. 2009). Also the short-term cross-linking and other modification of the lignin complex when wood is heat treated (Weiland and Guyonnet 2003, Tjeerdsma and Militz 2005) can essentially improve its strength properties, thus explaining the observed increments.

Even though many previous studies have indicated that temperature has a higher effect on the mechanical properties, the significant interaction observed in the analysis of some of the strength parameters indicates that temperature and duration need to be jointly regulated to optimize the final strength of the heat treatment product.

The findings of this study show that under dry process, a heat treatment condition of 149°C for 8 hours can be safely applied to pine, sweetgum, and oak without posing a significant negative impact on their essential mechanical properties. However, the strength requirement in the intended area of application should be strictly considered in determining the amplitude of the heat treatment process. For example, for flooring applications, it will be safe to treat the wood at 204°C for 5 hours' duration in order to increase the moisture repellency of the wood, given that the hardness and perpendicular compression are not significantly affected compared with the control treatment condition. It should also be noted that the high coefficient of variation observed in specimens treated at 204° C indicates higher variability in the strength properties of wood treated at this temperature. This might be explained by the weakening of the internal bonds within the structure of the wood as a result of the onset or extensive degradation of the lignin matrix and the hemicellulose constituents such that the response of the wood to applied load becomes more erratic and unpredictable. This causes a vast measure of unreliability concerning the strength-based consistency of such wooden structures while in service compared with the wood treated under the optimal conditions.

Shear strength appeared to be more negatively affected than the other strength properties, but it is probably the least evaluated and reported strength property; hence most previous studies have concluded that MOR is the most impaired strength property in heat-treated wood. Even though this finding might call attention to the need to monitor the shear strength of heat-treated wood because of its importance in constructional joints and fixtures, static bending is often considered by structural engineers to be a more critical property to closely evaluate and preserve because it is an indicator of wood behavior under stress in complex structural applications. With high temperature heat treating, these properties may be more closely related than normal. At high temperatures, shear strength is greatly reduced. Failure in internal shear would split the beam along the neutral plane causing a reduction in MOR.

This study provides a basis for product development and standardization in the North American wood heat treatment industry. This could pave the way for standard specification and grading of heat-treated wood.

The intent of this article is not to provide absolute values that are true for all tests using the same procedures. Between-tree variation would cause differences in actual strength values. The intent is to show the strength values so that the readers can ascertain the relationship between the strength value and duration and temperature of heat treating. Whether the values increase, decrease, or remain the same with increasing duration and temperature, the relationships (per species and strength test) should remain consistent for all tests using the same species and procedures.

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