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The Impacts of Heat Treatment on Lap Joint Shear Strength of Black Pine Wood

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This study was conducted to determine the impacts of heat treatment on lap shear strength, density, and mass loss of black pine wood. In the study, black pine wood boards bonded with polyurethane were subjected to temperatures of 160, 180, and 200°C for durations of 2 and 6 hours. Specimens having two layers were prepared from untreated and treated wood for mechanical testing of bond lines. Data were analyzed using variance analysis and Tukey's test to determine the impacts of changes in density and mass of heat-treated black pine wood on lap shear strength. The results indicated that the lap shear strength of black pine wood decreased as the intensity of heat treatment increased. The results also indicated that the minimum and maximum percentage decreases of lap shear strength were approximately 27% for 160°C and 2 hours and 78% for 200°C and 6 hours.

Keywords: Black pine; Density loss; Heat treatment; Lap shear strength; Mass loss

1. INTRODUCTION

In many European countries, the increased environmental pressures of the last few years have resulted in the important development of thermally modified wood as a non-biological alternative to classical preservation techniques [1]. The heat-treatment process for wood preservation is used as one of the alternatives to the use of chemicals for protecting wood. Heat-treated wood exhibits a lower affinity for water and a strongly modified wettability, which lead to important changes in its behavior with most coating or gluing processes [2,3].

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In the heat-treatment process, wood is heated to temperatures of 160 to 250°C; typically, heat-treatment temperatures exceed 200°C, depending on the species used and the properties of the material. Heating causes various changes in the structure of the wood. Initially, hemicelluloses begin to degrade, since they have the lowest molecular weights among the polymers in the wood. The degradation of hemicelluloses results in the reduction of OH bonds and the formation of O-acetyl groups. With the subsequent cross-link formation between the wood fibers, the wood becomes more hydrophobic. Heat treatment of wood is used primarily to increase durability, reduce hygroscopicity, and improve the dimensional stability of wood. The reduction of the ability of the wood to absorb water causes a decrease in the swelling and shrinkage of the wood, leading to improved dimensional stability [4]. In addition to these desirable changes, heat treatment also results in unfavorable effects, such as diminished strength and toughness [5–9].

Wood is composed of biopolymers such as cellulose, hemicelluloses, and lignin, as well as extractives and a small amount of inorganic components [10]. As a result of the numerous hydroxyl groups that occur in the individual main components of wood, it has a strong polar, *i.e.*, hydrophilic, character. The wettability of the surface of the wood, which is a prerequisite for the adhesion between substrate and adhesive, depends on a number of factors, such as the species, roughness and age of the surface, anatomical growth direction, penetration behavior, porosity, moisture content, hygroscopicity, chemical composition, and the pH of the wood [11–13].

Changes in chemical, physical, and structural properties of wood after heat treatment can affect the ability of adhesives to laminate the wood surface. The improved dimensional stability of heat-treated wood generally improves bonding performance, because stresses on the cured adhesive bond due to shrinking or swelling are reduced. However, heat treatment can be expected to cause significant changes related to adhesion, which makes it necessary to modify the bonding process.

Strong adhesion between the adhesive and the wood is possible if appropriate adhesive flow, penetration, wetting, and curing can be attained [14]. Heat-treated wood is less hygroscopic [15,16], and this can alter the distribution of the adhesive on the surface of the wood and the penetration of the adhesive into the porous wood structure. Several studies have shown that the wettability of wood with water decreases after heat treatment [2,3,14,17]. This effect occurs mainly because the surface of heat-treated wood is hydrophobic and less polar, and, therefore, it is significantly repellent to water. This might hinder waterborne adhesives from adequately wetting the surface. Generally, heat-treated wood is utilized in exterior applications, such as poolside

flooring and furniture, saunas, garden furniture, fencing, claddings, window frames, and doors. Heat-treated wood may also have potential for use in construction (e.g., for use as a structural element in the building industry). For a number of construction products, lamination is necessary, and exterior-type wood adhesives, which can withstand long-term wetting and drying, can be used to produce such products [14].

The goals of the current study were to determine the effects of heat treatment on lap shear strength, mass loss, and density loss of black pine wood and to display the effects of heat treatment on lap shear strength. Black wood pine was chosen for the study due to its availability in Turkey.

2. MATERIALS AND METHODS

Black pine (*Pinus nigra* Arn. *pallasiana* subsp. *pallasiana*) boards obtained from the Yenice Forests in Karabuk, Turkey, were used in the study to examine the effects of heat treatment on bonding performance of small, lab-scale glued samples, which were bonded with one structural, cold-setting adhesive. Polyurethane adhesives (Leim Marine Adhesive) were obtained from the Aktif Doga Company in Ankara, Turkey. Polyurethanes are in the class of compounds called reaction polymers, which includes epoxies, unsaturated polyesters, and phenolics.

A urethane linkage is produced by reacting an isocyanate group ($-N=C=O$) with a hydroxyl ($-OH$) group. Polyurethanes are produced by the polyaddition reaction of a polyisocyanate with a polyalcohol (polyol) in the presence of a catalyst and other additives [18]. These adhesives are widely used in high-resiliency, flexible-foam seating, rigid foam insulation panels, microcellular foam seals and gaskets, durable elastomeric wheels and tires, automotive suspension bushings, electrical potting compounds, high-performance adhesives and sealants, Spandex fibers, seals, gaskets, carpet underlay, and hard plastic parts [18]. For the current study, the boards had a thickness of 5 ± 1 mm, a width of 20 ± 2 mm, and a length of 150 ± 2 mm. Prior to heat treatment, the timber was dried in a kiln to the standard moisture content (MC) of 12% ($\pm 2\%$) using a conventional drying process at 30–40°C.

According to TS 642 [19], the black pine must be kiln dried in air until the moisture content is in the range of 11 to 13% before heat treatment. The samples were checked carefully for any defects, and clean samples were subjected to heat treatment. The test samples were subjected to heat treatment in a fully controlled oven with $\pm 1^\circ\text{C}$ sensitivity at three temperatures (160, 180, and 200°C) and two time periods (2 and 6 h) at atmospheric pressure. Untreated

(control) and heat-treated boards ($5 \times 150 \times 150$ mm) were then cut into slices ($5 \times 10 \times 150$ mm) and conditioned in a standard climate of 65% relative humidity (RH) at a temperature of 20°C . Then, two slices were bonded together into a small sample (Fig. 1).

The adhesive was applied with a brush at an application rate of 220 g/m^2 . The samples were pressed for 90 minutes in a hydraulic press at room temperature ($22 \pm 2^\circ\text{C}$) at a pressure of 1.0 MPa. Seven groups were bonded, consisting of six groups of heat-treated wood and one group of untreated wood (control) and ten replicates.

Small, clear black pine (*Pinus nigra* Arn. *Pallasiana* subsp. *pallasiana*) specimens ($20 \times 20 \times 30$ mm) were obtained from a local mill. Density measurements were carried out according to Turkish Standards (TS) TS EN 2472 [20].

$$d = \frac{m}{V} \text{ g/cm}^3,$$

where d is the density of the samples, m is the mass of the samples, and V is the volume of the samples.

According to TS EN 392 [21], lap shear testing must be conducted using a Zwick–Rowell 100-kN universal testing machine (obtained from Utest Company, Ankara, Turkey) equipped with a 2.5-kN load cell. Lap joint shear specimens were tested to failure at a cross-head speed of 3 mm/min, and lap shear strength was calculated by dividing the tension load by the area of overlap. Specimens were loaded until the onset of cracking. Lap shear strength measurements were carried out according to Turkish Standards (TS) TS EN 392 [21].

$$\tau = \frac{F_{\max}}{\text{Surface } (a \times b)},$$

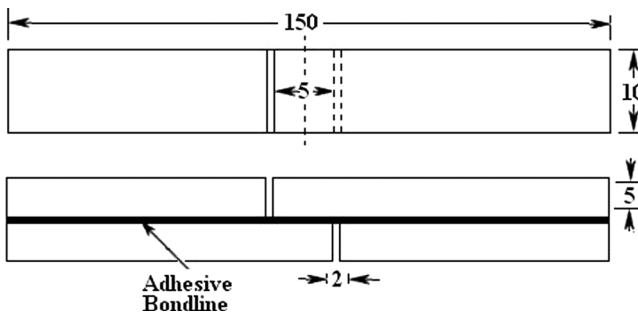


FIGURE 1 Samples used to measure lap shear strength (dimensions of the samples are in mm).

where τ is lap shear strength (LSS), F_{\max} is the tension load, a is the length of the specimens, and b is the width of the specimens.

3. RESULTS AND DISCUSSION

The results given in Table 1 were obtained using analysis of variance. All statistical calculations were based on a 95% confidence level. ANOVA tests indicated that all differences between control specimens and heat-treated specimens were significant. Also, a Duncan Test was conducted to determine the differences among groups. The results that were obtained indicated that the effect of heat treatment on lap shear strength was dependent on the treatment conditions. According to

TABLE 1 Change in Some Properties of Black Pine Wood After Heat Treatment

Heat treatment					
Temperature (°C)	Duration (hour)	Statistical values	LSS (N/mm ²)	Density (g/cm ³)	Mass loss (%)
Control	-	x	5998A	0.55A	-
		$\pm s$	572	0.02	-
		V %	10	3.64	-
160	2	x	4405B	0.54B	0.55A
		$\pm s$	349	0.015	0.02
		V %	8	2.76	3.63
	6	x	4023C	0.54B	0.75AB
		$\pm s$	464	0.023	0.04
		V %	12	4.24	5.33
180	2	x	2565D	0.54B	2.45C
		$\pm s$	313	0.011	0.03
		V %	12	2.03	1.22
	6	x	2392D	0.52C	2.75C
		$\pm s$	284	0.01	0.04
		V %	12	1.92	1.45
200	2	x	1666E	0.52C	3.40D
		$\pm s$	181	0.04	0.06
		V %	11	7.7	1.76
	6	x	1356E	0.51C	4.60E
		$\pm s$	204	0.01	0.07
		V %	15	1.96	1.52

X: Average, $\pm s$: Standard deviation, V: Coefficient of variation. Homogeneity groups: Same letters (A, B, C, and D) in each column indicate that there is no statistically significant difference among the samples according to the Duncan's multiple range test at $p < 0.05$. Comparisons were done between the control and test samples. Ten replicates were used in each test. All data in variance and one-way ANOVA tests were conducted at a confidence level of $p < 0.05$ (95%).

Table 1, the specimens treated at 160°C for 2 h had the minimum strength loss, whereas the specimens treated at 200°C for 6 h had the maximum strength loss.

The results show a logarithmic relationship between density loss and lap shear strength loss with a correlation coefficient of 79% ($r^2 = 0.79$). Hence, it can be suggested that density loss had a significant effect on lap shear strength (Fig. 2).

The relationship between mass loss and lap shear strength loss of black pine wood after heat treatment is demonstrated in Fig. 3. According to these results, as mass loss increases, there is a polynomial decrease in lap shear strength loss. Examining the correlation coefficient ($r^2 = 0.99$), it can be determined that there is a strong relationship between the increased mass loss and decreased lap shear strength.

Figure 4 shows the lap shear strength changes that occurred for the different durations of heat treatment. Lap shear strength loss was 27% at a heat-treatment temperature of 160°C for 2 h. As the treatment temperatures and durations increased, the strength of the specimens decreased. For example, at heat-treatment conditions of 200°C and duration of 6 h, there was a 78% reduction in the strength of the specimens.

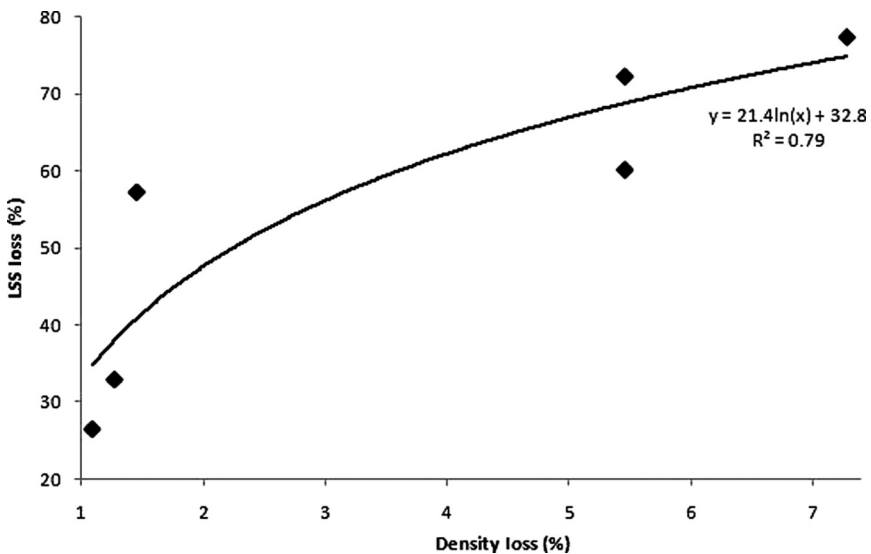


FIGURE 2 The effect of density loss on lap shear strength after heat treatment.

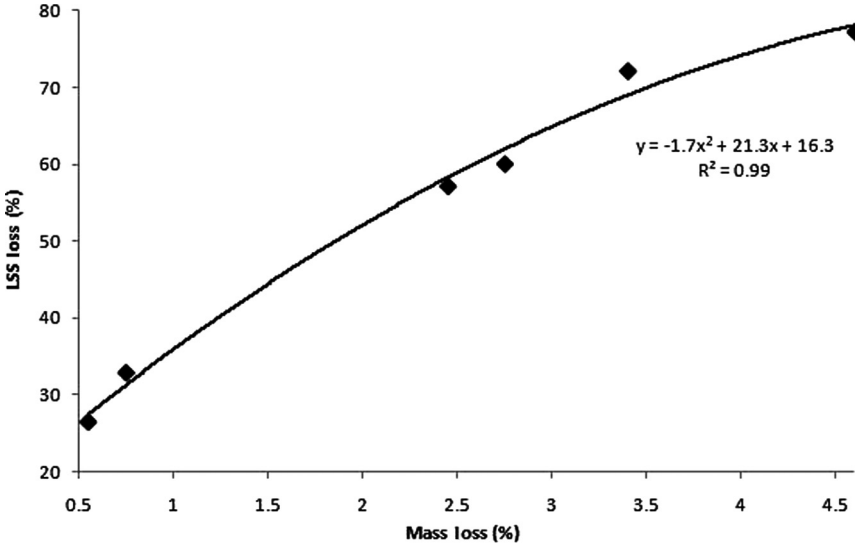


FIGURE 3 The effect of mass loss on lap shear strength loss after heat treatment.

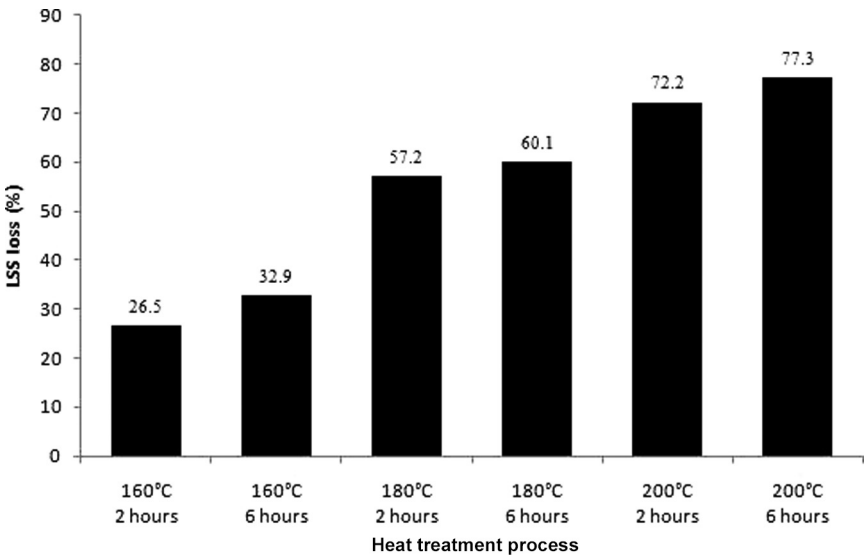


FIGURE 4 Changes occurring in lap shear strength according to heat treatment process.

As can be seen in Fig. 4, the differences in lap shear strength after treatment of the specimens at the same temperature but at different durations were significant; however, the magnitude of the effect of this variable was not as large as the effects that resulted from higher treatment temperatures. The higher temperature treatments resulted in significant reductions of the lap shear strengths of the specimens.

Contrary to the current findings, Follrich *et al.* [17] determined that there was no significant difference in bonding strength between the untreated reference and the heat-treated specimens. Additionally, within their three groups of heat-treated specimens, no significant differences were found. The percentage of wood failures, however, indicated clear differences. While no wood failures were observed in the untreated reference specimens, wood fractures occurred in 14, 47, and 59% of specimens that were heat treated for durations of 5, 30, and 45 min, respectively. These results indicate a progressive decrease in the mechanical strength of wood as the heat-treatment duration increases. The tension load (F_{\max}) measured at the onset of the first crack in the specimens was significantly higher in the heat-treated specimens compared with the untreated reference.

Similar effects of heat treatments were observed by Reiterer and Sinn [22], who determined that heat treatment affected the bonding and tension load of wood specimens depending on the adhesive system used for bonding [14].

4. CONCLUSIONS

In the current study, the effects of heat treatment on the lap shear strength of black pine were investigated. According to the test results, the mass and density losses that occurred during heat treatment significantly affected lap shear strength. This relationship was found to be parabolic and exponential. In addition, as the treatment conditions intensified, lap shear strength was affected to a greater extent, and it was found that treatment temperature also had stronger effects than the duration of the treatment. Therefore, it can be suggested that heat treatment significantly reduces bonding performance of wooden materials.

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