Properties of Thermally Treated Yellow Poplar, Southern Pine, and Eastern Redcedar

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Properties were evaluated for heat-treated yellow poplar (*Liriodendron tulipifera*), eastern redcedar (*Juniperus virginiana*), and southern pine (*Pinus echinata*) samples. Differences in discoloration, surface roughness, and hardness of the samples as a function of heat exposure were tested at temperature levels of 130 °C, 160 °C, and 190 °C. The experiments were carried out on defect-free eastern redcedar, yellow poplar, and southern pine samples with dimensions of 50 cm by 4 cm by 2 cm (longitudinal, radial, tangential) supplied by a local sawmill. A total of 80 samples, 20 for each temperature level, were used for the tests. Based on the findings, it appears that eastern redcedar specimens had the least discoloration values as compared to those of two other types of wood. In all cases, hardness values of the samples showed adverse influence of heat exposure. It seems that as temperature level increased, the surface quality of the samples from all three species was enhanced. All types of samples had significant discoloration as a result of heat treatment, and such findings were more prominent in the case of both pine and yellow poplar specimens. Overall hardness characteristics of the samples were adversely influenced due to heat exposure.

Keywords: Heat treatment; Surface roughness characteristics; Discoloration; Janka hardness

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INTRODUCTION

Wood as an organic material is susceptible to deterioration by environmental factors such as moisture, light, heat, and microorganisms. Therefore, wood and wood-based materials have certain disadvantages when they are designed to be used for exterior conditions. Thermal modification or heat treatment is an effective method to improve the dimensional stability and biological durability of wood.

One of the processes that is used to modify the properties of wood is heat treatment. This technique was developed in Europe during the early 1990s (Korkut and Guller 2008; Bakar *et al.* 2013). It is well known that heat treatment improves the dimensional stability and durability of wood (Militz 2002); heat treatment improves resistance against biological deterioration and reduces the equilibrium moisture content of wood (Metsä-Kortelainen *et al.* 2006). Although certain physical characteristics of wood can be enhanced, reduction in mechanical properties of the wood due to mass losses and thermal degredation is a major drawback of heat treatment (Korkut *et al.* 2008).

The other advantage of thermal modification is color modification of the final product. Interior designers use thermally modified wood in many projects. Changes in the color of wood can occur at ambient temperatures as a result of enzyme-mediated (Maillard) reactions between sugars, phenolic compounds, and amino acids. These reactions, which are similar to those that cause the browning of freshly cut fruit, occur in living parenchyma cells where they create amorphous globules of colored material (Yeo and Smith 2004). Thermal modification of wood causes hydrolysis and oxidation of the components (Thompson *et al.* 2007).

Mechanical properties of wood are influenced in various ways by thermal modification. Undesired side effects, in particular loss of strength and increased brittleness of the treated wood, have prevented a commercial utilization of thermal modification (Runkel and Witt 1953). Giebeler (1983) found a reduction of the modulus of rupture of wood of 20% to 50% after thermal treatment at 180 °C to 200 °C. Thermal modification of wood can increase its surface roughness, as shown by several studies (Gunduz *et al.* 2008; Korkut *et al.* 2008; Ayrılmıs *et al.* 2011).

In general, the color of the wood becomes darker with exposure to high temperature. The change of wood color depends on the heat treatment method and especially the treatment temperature and exposure time. The color stability resistance of the heat-treated wood exposed to outdoor conditions is better than that of natural wood. Color change of wood also takes place in the form of weathering when it is used in outdoor conditions (Mayes and Oksanen 2002; Gosselink *et al.* 2004; Aydemir and Gündüz 2009).

Three species of wood used in this work have economic importance in the furniture and construction industry in the USA. The physical and mechanical properties of these species have been well investigated, and there is a substantial amount of data in the literature (Korkut and Guller 2008; Ozcan *et al.* 2012; Kasemsiri *et al.* 2013). However, to our knowledge, there is little or no information on the properties of heat-treated poplar, southern pine, and eastern redcedar. Therefore the objective of this study was to evaluate color change, surface roughness, and hardness of the samples from these three species as a function of heat treatment.

EXPERIMENTAL

Materials and Sample Preparation

Defect-free eastern redcedar, yellow poplar, and southern pine samples with dimensions of 50 cm by 4 cm by 2 cm (longitudinal, radial, tangential) were supplied by a local sawmill. Sample dimensions for surface roughness tests were as follows: 12 cm by 4 cm by 2 cm. Sample dimensions for janka hardness and discoloration tests were as follows: 5 cm by 4 cm by 2 cm. After preparation, samples were kept in a conditioning chamber at 20 °C and relative humidity of 65% until they reached an equilibrium moisture content of 12%. The tangential surface of each sample was sanded sequentially with 100 and 200 grid sandpaper with application of several light strokes before heat treatment. A total of 80 samples, 20 for each temperature level of 130 °C, 160 °C, 190 °C, and as control samples were used for the experiments.

Thermal Modification Process

After stabilization of moisture content at 12%, samples were thermal heat treated in a laboratory type oven controlled at an accuracy of ±1 °C. Samples were heat treated at 130 ºC, 160 ºC, and 190 ºC for 8 h. After the heat treatment process, all samples were kept at room temperature for 2 h. Five replicates were used for each treatment condition regarding time and temperature. After the heat treatment, the samples were taken from the oven for the colorimetric evaluation.

After each heat treatment application, the samples were weighted and measured (length, width, and thickness) for later calculation of density.

Roughness Measurement of the Samples

The stylus method is a widely used technique to measure surface roughness of different materials resulting in quantitative numerical values on the surface of a sample. The profilometer has a skid-type diamond stylus with 5 mm tip radius and 15.2 mm span on a surface. Technical details and working principles of stylus type profilometer are presented by Ulker (2018). Roughness parameters such as average roughness (R_a), mean peak-to-valley height (R_z), and maximum roughness (R_{max}) can be calculated from the digital information obtained from the surface (Ulker and Hiziroglu 2017).

Samples with the size of 12 cm, 4 cm by 2 cm (longitudinal, radial, tangential) were used for random roughness measurements. A total of five samples were used for roughness measurements of the groups as illustrated in Fig. 1. In each tree specimen, a total of 20 samples were used for roughness measurement.

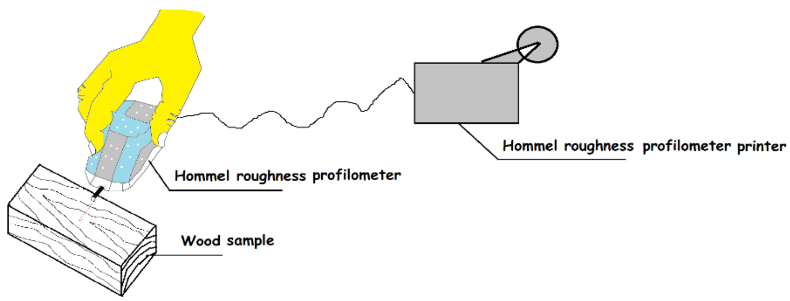

Fig. 1. Roughness test set-up

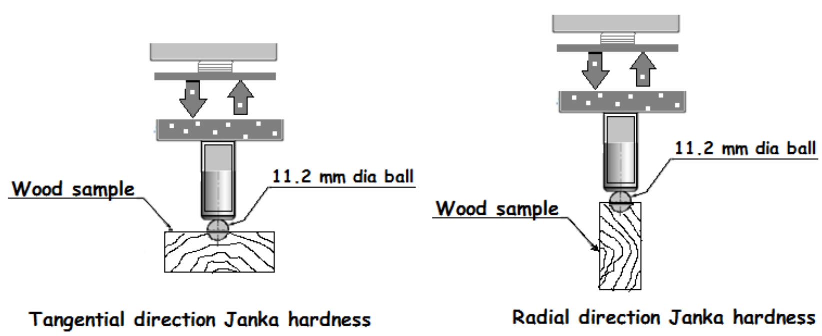

Fig. 2. Janka hardness test set-up

Janka Hardness Test of the Samples

Hardness values of defect-free eastern redcedar, yellow poplar, and southern pine samples were determined by embedding a hemisphere steel having 11.2 mm diameter onto their tangential and radial surfaces using a Comten 95 Series Universal Testing machine. Five measurements on each grain orientation were taken from each sample and recorded in kg to evaluate their Janka hardness, as illustrated in Fig. 2 (Ulker 2018).

Discoloration Evaluation of the Samples

The lightness data were collected before and after heat treatment, and lightness measurements were taken from the surface of the samples by using a colorimeter FRU WR-10QC. The colorimeter heat sensor was 8 mm in diameter. Measurements were carried out using a D 65 illuminant and 10-degree standard observer. Percentage of reflectance, collected at 10 nm intervals over the visible spectrum ranging from 400 to 700 nm was converted into the CIELAB color system, where L^* describes the lightness, whereas a^* and b^* describe the chromatic coordinates on the green-red and blue yellow axis, respectively. From the $L^*a^*b^*$ values, the difference in the lightness (ΔL^*) and chroma coordinates (Δa^* and Δb^*), hue angle (h), saturation (C^*) and total color difference (ΔE) were calculated using the following formulae.

$$\Delta L^* = L^*_t - L^*_c \qquad h = \text{arctg}(b^*/a^*)$$

$$\Delta a^* = a^*_t - a^*_c \qquad C^* = (a^{*2} + b^{*2})^{1/2}$$

$$\Delta b^* = b^*_t - b^*_c \qquad \Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$$

where L^*_t a^*_t b^*_t are L^*, a^*, and b^* of the heat treatment samples; L^*_c a^*_c b^*_c are L^*, a^* and b^* of the control samples, respectively.

To analyze the significant differences between all the parameters used in this study, multiple analysis of variance (MANOVA) was performed. In order to compare the groups, the Duncan range test was employed. Finally, all results found in the work were evaluated using IBM Statistical Package for the Social Sciences (SPSS) software.

RESULTS AND DISCUSSION

In this study, two different types of hypotheses were evaluated. H_0: There is no significant change in surface roughness and color change during the heat treatment at different temperature levels (130 °C, 160 °C, and 190 °C) with three types of wood species. H_1: Color changes and surface roughness could exist after heat treatment at the three different temperature levels of 130 °C, 160 °C, and 190 °C.

Surface Roughness Test

Statistical values of surface roughness of eastern redcedar, yellow poplar, and southern pine are given in Table 1. Overall, surface roughness levels of the samples decreased substantially with increasing temperature level. Control samples of yellow poplar, eastern redcedar, and southern pine had R_a values of 11.32 μm, 7.68 μm, and 3.76 μm, respectively. These values decreased to 10.09 μm, 7.35 μm, and 3.41 μm, respectively, when the samples were exposed to a temperature of 130 °C. Exposure of the samples to 160 °C and 190 °C temperature levels further decreased their average roughness values, according to the R_z and R_{max} values (Fig. 3).

Table 1. Average Density and Surface Roughness Values of Heat Treated Samples (Numbers in parenthesis are standard deviation values)

Species and Temperature Level		Density (g/cm³)	Surface Roughness Values		
			R_a (µm)	R_z (µm)	R_{max} (µm)
Yellow Poplar	Control	0.45 (0.06)	11.32 (0.61)	66.41 (7.15)	79.98 (5.41)
	130°C	0.44 (0.06)	10.25 (0.79)	50.38 (10.98)	75.33 (13.62)
	160°C	0.43 (0.05)	10.09 (0.59)	44.42 (8.49)	53.31 (7.46)
	190°C	0.41 (0.06)	4.88 (0.51)	41.42 (8.49)	50.62 (4.33)
Eastern Redcedar	Control	0.49 (0.07)	7.68 (0.61)	54.73 (3.20)	82.56 (5.22)
	130°C	0.47 (0.06)	7.35 (0.72)	49.91 (4.49)	62.46 (7.08)
	160°C	0.46 (0.06)	6.98 (0.96)	44.58 (6.29)	53.55 (8.50)
	190°C	0.45 (0.05)	6.75 (1.11)	43.58 (5.68)	49.96 (5.51)
Southern Pine	Control	0.62 (0.06)	3.76 (0.51)	29.9 (4.41)	56.93 (3.48)
	130°C	0.61 (0.05)	3.41 (0.36)	27.16 (5.57)	52.68 (5.26)
	160°C	0.59 (0.05)	2.91 (0.64)	23.64 (4.15)	35.26 (3.95)
	190°C	0.57 (0.05)	1.61 (0.42)	18.60 (2.53)	28.09 (4.84)

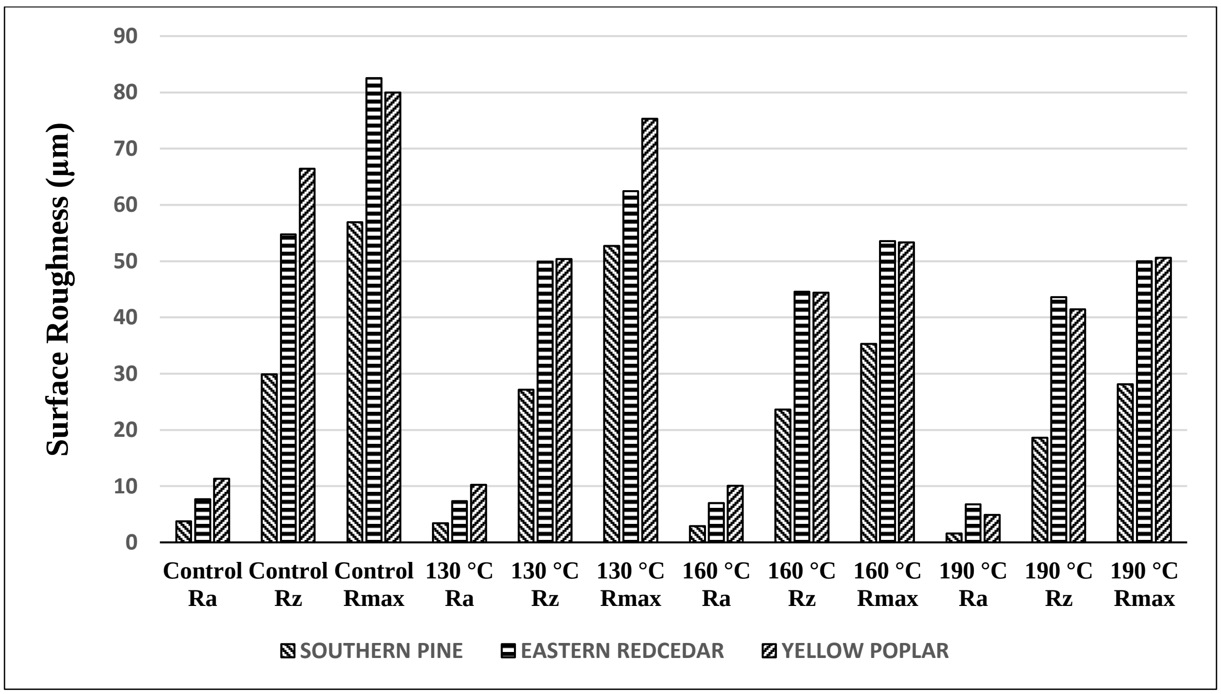

Fig. 3. Surface roughness of samples

Based on the statistical analysis, significant enhancement of roughness characteristics of the control samples of three species were determined when they were exposed to a temperature of 190 °C. Overall roughness values of the samples showed a significant improvement when heat exposure temperature levels were increased sequentially 130 °C, 160 °C, and 190 °C. In a past study, major change in roughness of various types of wood species were observed when the samples were exposed to a temperature of 200 °C for 8 h (Trisna and Hiziroglu 2013). Anatomical structure of each species also had a certain amount of influence on their surface roughness values. Statistical analysis related to the effectiveness of the temperature on surface roughness of the samples, are displayed in Table 2.

Table 2. Analysis of Variance Related to the Effect of Average Surface Roughness Values

Applied Tests	Mean Square	F-value	Level of Significance ($p \leq 0.05$)
Temperature	57.88	126.73	0.000
Wood Species	405.05	886.87	0.000
Temperature x Wood Species	18.03	39.49	0.000

Based on statistical analysis, significant differences were observed between surface roughness characteristics, wood species, and heating levels at a 95% confidence level. The heat exposure process with different wood species was found to be effective ($p < 0.05$) on surface roughness parameter values (R_a, R_z, and R_{max}) and wood species.

Table 3. Comparative Test Results for Average Surface Roughness (R_a) Values and Homogeneity Groups

Parameters	Groups	H.G.* *A*	H.G.* *B*	H.G.* *C*
Temperature	Control	7.591	****	****
	130°C	****	7.004	****
	160°C	****	6.662	****
	190°C	****	****	4.416
Wood Species	Yellow Po.*	****	****	9.140
	Eastern Rc.*	****	7.194	****
	Southern P.*	2.920	****	****

* Homogeneity groups, Po.* poplar, Rc.* Redcedar, P.* pine

Table 3 also displays the Duncan test results related to the homogeneous subsets according to the values determined in this work. Homogeneity group values-A of 7.591 µm and 2.920 µm were determined for heating levels, wood species, respectively.

Both wood species samples had higher surface roughness values in control samples except the southern pine control sample. It appears that heat exposure affected surface quality of wood species. In Figs. 4, 5, and 6, relationships between surface roughness parameters and heat levels are illustrated.

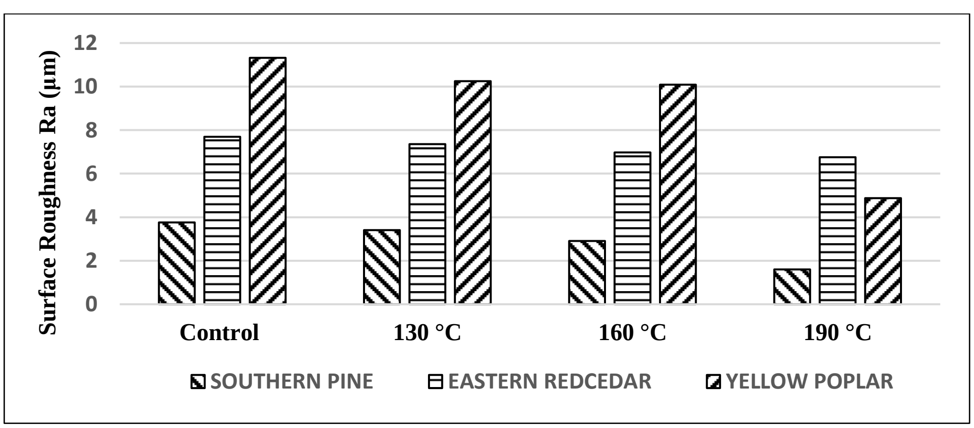

Fig. 4. Average surface roughness values (R_a) of the samples

As heat levels increased, all average surface roughness values (R_a) of wood species decreased. Specifically, southern pine had the greatest decrease with a value of 57.4%, followed by yellow poplar 56.9%, and finally eastern redcedar the least 12.1% increase. If the trends are compared between the average surface roughness (R_a) of species, yellow poplar had a steeper slope than that of other species.

Evaluation of Janka Hardness Test Result of Samples

Some of the samples tested in this work even crumbled and collapsed during the hardness test. In general, hemicellulose in the cell wall is degraded due to heat, resulting in a reduction in strength characteristics of the samples. By increasing the temperature above 200 °C, the thermal degradation of cellulose and formation of volatile products proceeds rapidly (Manninen *et al.* 2002). After heat treatment, lignin shows evidence of thermal degradation. Relatively weak aliphatic bonds break down and hydrocarbon fragments are released. As secondary reactions, hydrocarbon fractions are degraded further and repolymerization occur (Avni *et al.* 1985).

All three types specimens had almost 50% reduction in their hardness values when they were exposed to a temperature of 190 °C as compared to that of control samples. Statistically significance of hardness values of the samples exposed to three temperature levels was clearly determined and illustrated in Table 4.

Table 4. Janka Hardness Values of Heat Treated Samples (Numbers in parenthesis are standard deviation values)

Species and Temperature Levels		Hardness Values (kg)	
		Radial	Tangential
Yellow Poplar	Control	644.09 (34.84)	525.00 (28.07)
	130 °C	537.27 (96.52)	469.55 (64.8)
	160 °C	414.09 (50.90)	383.18 (66.94)
	190 °C	326.36 (46.03)	275.45 (68.15)
Eastern Redcedar	Control	385.45 (80.59)	353.64 (68.66)
	130 °C	360.00 (53.96)	310.00 (61.32)
	160 °C	281.82 (78.13)	245.45 (46.93)
	190 °C	192.73 (42.83)	170.45 (40.96)
Southern Pine	Control	328.18 (21.18)	279.09 (37.58)
	130 °C	276.36 (57.59)	253.64 (54.03)
	160 °C	226.82 (55.76)	210.45 (70.04)
	190°C	156.36 (35.82)	143.18 (30.47)

Both wood species samples had higher surface roughness values at control samples except the southern pine control group. It appears that heat exposure reduced the hardness of wood species.

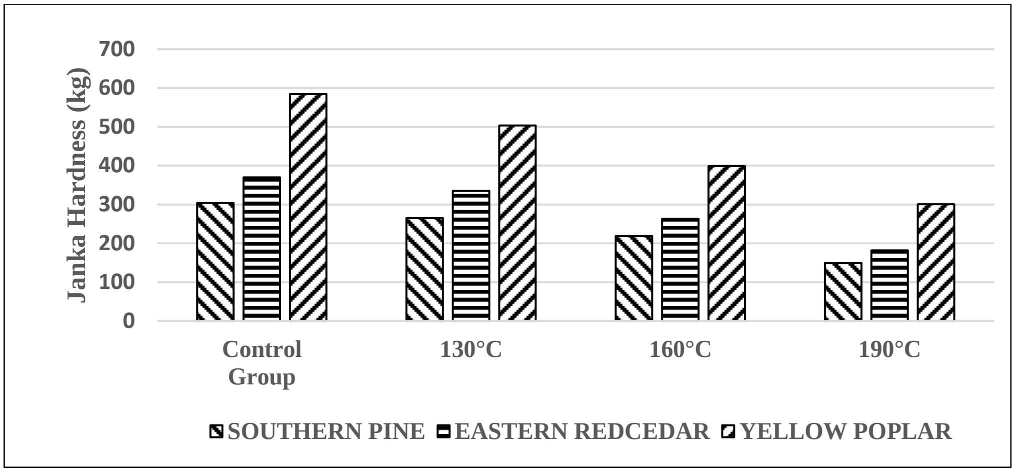

Fig. 5. Janka Hardness of samples at different temperature

As illustrated in Fig. 5, it can be concluded that after heat treatment, all Janka hardness values of the specimens decreased. Specifically, southern pine samples exposed to 190 °C had the minimum Janka hardness value of 146.8 kg. The highest average value of 644.1 kg was determined for control samples of yellow poplar followed by eastern redcedar and southern pine. Among three species, yellow poplar has the highest density value of 510 kg/m^3. It is clear that heat exposure adversely affected overall hardness of all three species. In a previous study, samples of mindi (*Melia azedarach* L.), mahogany (*Swietenia macrophylla*), red oak (*Quercus falcata* Michx.), and Southern pine (*Pinus taeda* L.) exposed to heat treatment had lower Janka hardness values (Kasemsiri *et al.* 2009).

It is an accepted fact that heat treatment adversely influences overall mechanical properties including hardness. Several past studies also confirmed this (Ulker *et al.* 2012; Percin *et al.* 2015). Heat treatment also darkens almost any kind of wood species. Statistical values of color values of wood samples are given at Table 2. Overall color levels of the samples decreased substantially with increasing temperature. In general the magnitude of darkening of the sample increased with rising of temperature (Andromachi and Barboutis 2014). An average lightness value of 72.4 was found for southern pine control samples, followed by yellow poplar samples. Eastern redcedar control samples having mostly hardwood had a relatively lower *L* value (51.8) than the other two species.

The non-heat treatment samples had 30% higher Janka hardness values than those of control samples. From this, it can be concluded that heat treatment adversely influenced overall hardness values of the samples.

Evaluation of Discoloration Test Result of Samples

Heat exposure affected lightness values of wood species. All average lightness values (*L*) of species decreased with increasing temperature. Specifically, yellow poplar had the greatest decrease with a value of 54.9%, followed by southern pine 46.0%, and finally eastern redcedar the least 36.6% value. Therefore, it appears that an increase in temperature levels adversely influenced all lightness values for samples.

Table 5. Discoloration Values of Heat Treated Samples (Numbers in parenthesis are standard deviation values)

Species and Temperature Levels		Color Characteristics		
		L	***a***	***b***
Yellow Poplar	Control	72.38 (3.56)	2.93 (0.62)	19.03 (3.30)
	130 °C	68.82 (2.10)	3.34 (1.12)	19.38 (3.21)
	160 °C	57.10 (4.36)	7.28 (1.52)	23.00 (2.71)
	190 °C	32.56 (2.66)	6.79 (1.22)	12.14 (2.57)
Eastern Redcedar	Control	51.80 (4.95)	11.57 (1.99)	21.29 (2.56)
	130 °C	47.15 (5.49)	11.66 (1.63)	20.05 (2.87)
	160 °C	42.73 (4.28)	10.68 (0.84)	19.08 (2.13)
	190 °C	32.82 (2.03)	8.46 (0.97)	14.55 (1.24)
Southern Pine	Control	70.99 (4.99)	5.72 (0.70)	21.43 (2.16)
	130 °C	68.85 (5.75)	5.70 (0.76)	22.40 (3.12)
	160 °C	55.49 (7.24)	9.40 (2.10)	26.16 (3.46)
	190 °C	38.34 (5.20)	9.12 (1.37)	18.18 (4.80)

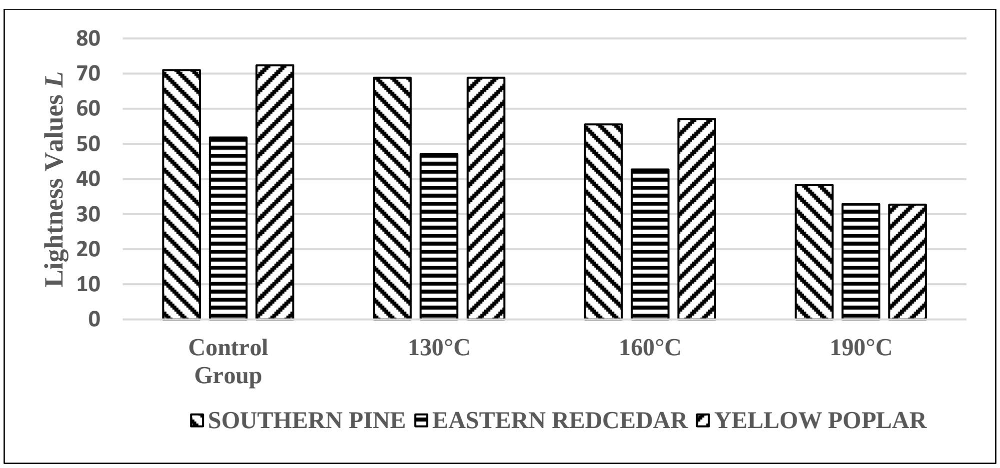

Fig. 6. Average Lighting values (*L*) of samples at different temperature

Variance analysis of the effectiveness of the heating level on lightness levels of the samples is displayed in Table 6.

Table 6. Analysis of Variance Related to the Effect of Lightness Level (*L*) Based on the Temperature, Grain Orientation, and Wood Species

Applied Tests	Mean Square	F value	Level of Significance ($p \leq .05$)
Temperature	8402.98	731.46	0.00
Wood Species	4179.40	363.80	0.00
Grain Orientation	447.00	38.91	0.00
Temperature x Wood Species	415.49	36.16	0.00
Temperature x Grain Orientation	22.11	1.92	0.81
Wood Species x Grain Orientation	183.05	15.93	0.00
Temperature x Wood Species x Grain Orientation	13.45	1.17	0.309

Based on statistical analysis, significant differences between wood species, cutting direction, and heating levels were observed at a 95% confidence level. Heat exposure process with different wood species was found to be effective ($p < 0.05$); however heat exposure and grain orientation were not effective.

Table 7 also displays the Duncan test results related to the homogeneous subsets according to the values determined in this work. Homogeneity group values A of 34.57, 43.62 and 50.17 were determined for heating levels, wood species and grain orientation respectively.

Table 7. Comparative Test Results for the Effect of Heat Exposure Levels on Various Properties of the Wood Samples for Homogeneity Groups

Parameters	Groups	H.G.* A	H.G.* B	H.G.* C	H.G.* D
Temperature	Control	****	****	****	65.06
	130 °C	****	****	61.60	****
	160 °C	****	51.77	****	****
	190 °C	34.57	****	****	****
Wood Species	Yellow P.*	****	57.72	****	****
	Eastern Rc.*	43.62	****	****	****
	Southern Pi.*	****	58.42	****	****
Grain Orientation	Cross S.*	50.17	****	****	****
	Tangential S.*	****	54.24	****	****
	Radial S.*	****	55.35	****	****

* Homogeneity groups, P* Poplar, Rc* Redcedar, Pi* Pine, S* Section

Discoloration in the form of darkening of wood during heat exposure is a result of chemical changes in the cell wall of polymers. Statistical analyses revealed that significant differences existed among the L^* values of the samples exposed to different temperature levels as well as control samples. Yellow poplar and southern pine samples showed relatively similar levels of discoloration values in contrast to eastern redcedar. As stated previously, eastern redcedar samples having mostly hardwood portion with reddish color would be the reason for such a finding.

In further studies, it would be desirable to evaluate dimensional behavior of heat-treated species as they are exposed to various levels of relative humidity in order to gain a better understanding their behavior.

CONCLUSIONS

The following concluding remarks can be drawn from this study:

1. Based on the findings this work, samples from all three species (yellow poplar [*Liriodendron tulipifera*], eastern redcedar [*Juniperus virginiana*], and southern pine [*Pinus echinata*]) resulted in significant discoloration as a function of heat exposure.
2. Color change of the samples was more prominent in the case of pine and yellow poplar species.
3. It seems that heat treatment did not substantially affect overall surface roughness of the samples.
4. Similar to the findings of previous studies, heat treatment adversely influenced mechanical properties, and in this work hardness of the samples reduced heat exposure.

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