

Characteristics of Hot-compressed Poplar Wood Boards

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The influence of thermal modification by hot-compressing was investigated relative to the physical, mechanical, anatomical, crystallinity, and colour characteristics of poplar wood boards. The boards were modified by a hot-compressed method under various temperature stages. The physical and mechanical properties of hot-compressed poplar wood increased with increased pressing temperature. Likewise, the highest crystallinity index (68.7%) of X-ray diffraction (XRD) analyses was found in the samples pressed at 210 °C. Microscopic investigation, revealed that there were some structural deformations in early and late wood, annual ring, *etc.*, of the compressed samples at 170 °C, 190 °C, and 210 °C. In a colour measurement test, it was determined that samples had different colour values in terms of temperature increase. The results achieved in this study demonstrated that the physical and mechanical properties of hot-compressed boards improved with increased press temperature. As a result, a thermal compression method could be preferred to advance end-usage features of low-density wood materials produced from fast-growing tree species like poplar, Douglas fir, spruce, yellow pine, eucalyptus, *etc.*

Keywords: Poplar wood; Thermal modification; Hot-compressed boards

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INTRODUCTION

The thermal modification of wood and wood boards is thought to advance its dimensional durability and tendency to fade (Yeo *et al.* 2010). Different heat processes, such as the ThermoWood® process, retification process, oil-heat treatment processes, Plato process, and Bois Perdure, are preferred in the wood products sector (Militz 2002). Physical and mechanical characteristics of wood materials could be enhanced with a thermal modification method. Seborg *et al.* (1945) described a process called Staypak, which involved pressing and thermal treatment. Tarkow and Seborg (1968) conducted experiments on wood surface density. After the 1980s, due to their low density and cost, thermally modified wood boards achieved great market success in Asia (Norimoto 1994; Wang *et al.* 2000). The thermally modified woods display improved physical and mechanical characteristics. To illustrate, thermally modified wood materials can be utilized in architectural construction, furniture production, flooring materials, roof panelling, interior and exterior planking of structures, light and sound barrier, and window and door carpentry (Korkut *et al.* 2008; Korkut and Kocaefe 2009).

A lot of research has been conducted on wood features, such as dimensional stability, bending characteristics, anatomical properties, drying characteristics, termite, insect, fungi and decay resistance, moisture desorption and absorption, surface corrosion strength, shearing strength, surface toughness, surface quality, specific gravity, and nail/screw withdrawal strength (Blomberg *et al.* 2005; Wang and Cooper 2005; Unsal

and Candan 2007; Unsal and Candan 2008; Welzbacher *et al.* 2008; Abraham *et al.* 2010; Dogu *et al.* 2010; Candan *et al.* 2013a, 2013b; Gao *et al.* 2016).

It has been observed that the density of produced materials tends to be high near the surface, while it is low in the internal parts in various press temperature and time-based studies. The density of wood boards changes according to pressure parameters, wood dampness, temperature, *etc.* These changes improve the physical and mechanical properties of wood boards (Wang and Winistorfer 2000; Candan 2007; Candan *et al.* 2013b). A thermal modification treatment may influence aspects such as the drying features, density, dimensional stability, surface quality, and Janka hardness. Unsal and Candan (2008) researched the physical and mechanical features of pine wood and found that the moisture content diminished, whereas the Janka hardness and density rose. Unsal *et al.* (2009) conducted thermal modification with high pressure on pine wood boards. They revealed that the thickness swelling values of the boards improved with increasing temperature and pressure. Unsal *et al.* (2003) investigated the influences of thermal modification on the mechanical and physical features of eucalyptus wood. As a result of this research it was found that the swelling, Janka hardness, and density values decreased once the temperature increased.

There have been numerous studies discussing the anatomical properties of thermally compressed wood boards (Boonstra *et al.* 2006; Dogu *et al.* 2010; Awoyemi and Jones 2011; İçel and Şimşek 2016). These studies focus on the effects on the anatomical properties of wood boards treated at different pressures, temperatures, and times.

When literature studies are examined, it has been found that there are some changes in the chemical properties of thermally modified wood boards. Boonstra and Tjeerdsmā (2006) determined that as temperature increased, extractives and lignin contents increased, whereas holocellulose, alpha cellulose, and hemicellulose contents decreased because of depolymerisation of cell wall components and degradation of holocellulose. Alén *et al.* (2002) found similar results. They revealed that lignin and extractives contents increased, but carbohydrates decreased with increasing of temperature. Yildiz *et al.* (2006) noticed that cellulose and hemicellulose contents were diminished; however, lignin content increased in terms of chemical composition of thermal modified wood samples. In another study, González-Peña *et al.* (2009) investigated the effects of heat on the chemical composition of thermal modified beech, pine, as well as spruce woods and they determined lignin content increased, while cellulose content reduced depending temperature increase.

According to the results of studies in the literature, it is seen that the physical, mechanical, anatomical, and chemical properties of wood can permanently change in temperatures over 150 °C. In this research, the anatomical, physical, and mechanical characteristics of thermally modified poplar wood boards under different temperatures were examined.

EXPERIMENTAL

Materials

The wood samples used in this study were supplied from the Department of Forestry in Kastamonu, Turkey. Wood samples were obtained from different hot-compression processes. Defect-free poplar wood (*Populus* spp.) solid panels with

dimensions of 25 mm × 100 mm × 500 mm were hot-compressed using a laboratory hot press at 130 °C, 150 °C, 170 °C, 190 °C, and 210 °C and under 1 atm pressure for 45 min. The panels were pre-dried to a moisture content of 14.7% before hot pressing; after this process the average moisture content of the hot compressed panels decreased to 7.2%. A total of 36 boards, 6 for each treatment group, were used.

Methods

Mechanical properties

Control and hot-compressed samples were cut into 20 mm × 20 mm × 300 mm and 20 mm × 20 mm × 30 mm pieces for determining their mechanical properties. Compression tests, bending tests, and modulus of elasticity were performed according to Turkish Standards Institute (TSE) TS 2595 (1977), TSE TS 2474 (1977), and TSE TS 2478 (1978). In addition, all mechanical experiments were performed at Kastamonu University, in the Forest Faculty, Wood Chemistry and Wood Mechanic Labs. In mechanical experiments, a universal test machine was used (Shimadzu Corporation, Kyoto, Japan). A compression test was performed at 1.5 mm/min at room temperature and at breaking force. The bending test was performed at 4 mm/min and at room temperature conditions. The load was applied to radial face of the test samples in the tangential direction of the annual rings. Samples of bending strength tests were used to determine the modulus of elasticity in bending (Bozkurt and Göker 1996).

X-ray diffraction (XRD) analysis

XRD analysis was performed on samples in the form of wood powder. The control and hot-compressed wood boards were cut into small pieces and were milled in a Wiley Mill (Swedesboro, NJ, USA). Then, XRD analyses were performed with these powders.

X-ray powder patterns were measured using a Bruker D8 Advance Spectrometer (Billerica, MA, USA). The diffractometer was equipped with a two circle (θ and 2θ) goniometer housed in a radiation safety enclosure. The X-ray source was a sealed 2.2 kW Cu X-ray tube, maintained at an operating current of 40 kV and 30 mA. The goniometer was computer controlled with independent stepper motors and optical encoders for the θ and 2θ circles with the smallest angular step size of $0.0001^\circ 2\theta$. The samples were scanned in the range of 5° to $60^\circ 2\theta$. A step size of 0.02° and a step time of 1.0 sec were used during the measurements. A Peltier cooled solid-state [Si(Li)] detector (Sol-X) with a useful energy range of 1 KeV to 60 KeV was used as the detector. No correction was made for $K\beta$ radiation. A set of 2° Soller slits were used to lower the horizontal beam divergence.

Anatomical characteristics

To perform anatomical analyses, after hot-compression and air-conditioning, cubic samples (2 mm on a side) were obtained from the wood panels. The cubes were softened by boiling for 6 h/d during 5 d. Thin samples were taken from cross-sections of the cubes by using a Thermo Shandon Finesse 325 microtome (Thermo Fisher Scientific, Waltham, MA, USA) and the slides were prepared. The slides were stained with safranin to supply a good contrast between the early- and late-woods and thus to obtain clear observations of changes in the wood structure. The images were observed under a Leica DM3000 light microscope (Leica microsystems, Wetzlar, Germany).

Colour measurement

The colour wood samples were measured using a Konika Minolta CM-2500-d series spectrophotometer (Konika Minolta, NJ, USA). The CIELAB colour system consists of three parameters, L^* , a^* , and b^* . The L^* axis represents the lightness and varies from 0 (black) to 100 (white). The symbol $+a^*$ is for the red, $-a^*$ for green, $+b^*$ for yellow, and $-b^*$ for blue. Total colour change ΔE^* was calculated using the L^* , a^* , and b^* data of each sample. Equation 1 was used for this purpose.

$$\Delta E^* = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2} \quad (1)$$

Data analysis

The obtained data were statistically analysed with SPSS (IBM, 23 software, New York, NY, USA). Both analysis of variance (ANOVA) and Duncan test were conducted. Homogenous groups were located according to the Duncan test, where factor effects were significant with a 0.05% error rate.

RESULTS AND DISCUSSION**Physical Properties**

The amounts of reduction in the thickness and density of thermally compressed wood samples are shown in Table 1.

Table 1. Thickness Reduction and Density of Hot-compressed Wood Samples

Temperature (°C)	Decrease in Thickness (%)	Density (g/cm ³)
Control	Non-compressed	0.379
130°C	4.44	0.456
150°C	11.04	0.466
170°C	10.71	0.471
190°C	19.07	0.489
210°C	28.96	0.533

As the press temperature increased under pressure, a reduction of up to 29% in the thickness of wood samples was observed. The density of poplar wood increased by up to 29% with increased press temperature under pressure. Similar findings were obtained by Unsal and Candan (2008) and Candan *et al.* (2013b). This study showed that as press temperature increased, the mean and peak density increased and the thickness of the compressed wood samples decreased.

Mechanical Properties

As a result of this study, by the time wood boards were thermally modified, it was observed that the physical and mechanical characteristics of boards improved. The results of the ANOVA and Duncan tests for mechanical features are shown in Table 2.

Upon examining the chart results, it was observed that the resulting values of all mechanical tests increased in stepwise manner, dependent upon the press temperature. After the literature was investigated, it was seen that the thermal modification enhanced the physical and mechanical properties of the boards. Boonstra *et al.* (2007) determined that there is a small decrease (3%) in modulus of rupture and a significant increase (28%)

in compression strength after heat treatment in wood board samples. The modulus of elasticity increased approximately 26% after heat treatment. Inoue *et al.* (1993) determined 20%, 45%, and 80% increases in the modulus of rupture of the samples treated at 180 °C, 200 °C, and 220 °C for 8 h, respectively.

Table 2. Results of Compression Strength, Modulus of Elasticity, and Modulus of Rupture Tests

Samples	Compression Strength (N/mm ²)	Bending Strength (N/mm ²)	Modulus of Elasticity (N/mm ²)
Control	22.13 (0.35) a	52.29 (4.16) a	6844.23 (393.09) a
130 °C	25.72 (8.60) a	54.45 (1.78) ab	7141.29 (148.89) a
150 °C	39.55 (7.17) b	62.54 (7.53) bc	7536.73 (560.78) ab
170 °C	47.43 (4.41) c	67.21 (2.04) c	8251.3 (640.89) b
190 °C	56.03 (3.68) d	76.44 (13.51) d	9617.17 (857.50) c
210 °C	67.86 (3.03) e	82.82 (1.48) d	10868.03 (548.96) d

$p \leq 0.05$, values in parentheses are standard deviations; ^{a,b,c,d,e} values with the same letter are not significantly different (Duncan Test)

Candan *et al.* (2013a) state that the boards pressed at 2 MPa and at 150 °C had maximum thickness swelling values. They explained that thermally compressed samples with 2 MPa at 170 °C had the highest density values. In another study, Candan *et al.* (2013b) noticed that the vertical density profile values of thermally compressed boards were higher than the values of the controls. Cloutier *et al.* (2008) saw that densification was the result of wrinkling in the cell walls after heat compression. They determined that the oven-dry density rose from approximately 374 kg/m³ to 924 kg/m³. Furthermore, with respect to mechanical properties, these researchers found that the modulus of elasticity in tension and bending of thermally modified boards was approximately twice as much as for the control samples. It was noticed that the Brinell hardness rose from 17 MPa for the control to 45 MPa for thermally modified wood at 200 °C.

Anatomical Characteristics

Microscopic analyses were conducted in cross-sections of the control and hot-compressed samples to understand the effects of hot pressure on the anatomical structure of the wood. Images of the untreated wood samples are demonstrated in Fig. 1a. Collapses and disintegrations were seen in the lumen and cell walls of the earlywoods of hot-pressed samples at 170 °C, 190 °C, and 210 °C. Furthermore, it was distinguished that rays zigzagged in the earlywoods of these samples. Because latewood is denser and tighter than earlywood, this did not occur in this zone. The disintegration of lumens in earlywood caused a wavy structure in this zone. However, the latewood of hot-compressed samples remained smooth. No disintegrations were noticed in the control samples and the samples treated at 130 °C and 150 °C, and very few deformations occurred in the samples pressed at 130 °C and 150 °C. Dogu *et al.* (2010) found the same results in their anatomic research conducted on thermally compressed Scots pine (*Pinus sylvestris*) panels, and they determined that the anatomical changes in earlywood and latewood zones were connected to temperature and pressure. Likewise, İçel and Şimşek (2016) used ash (*Fraxinus excelsior*) and spruce (*Picea abies*) in their research, and they confirmed that hot-pressing caused deformations in the anatomic structures of wood boards.

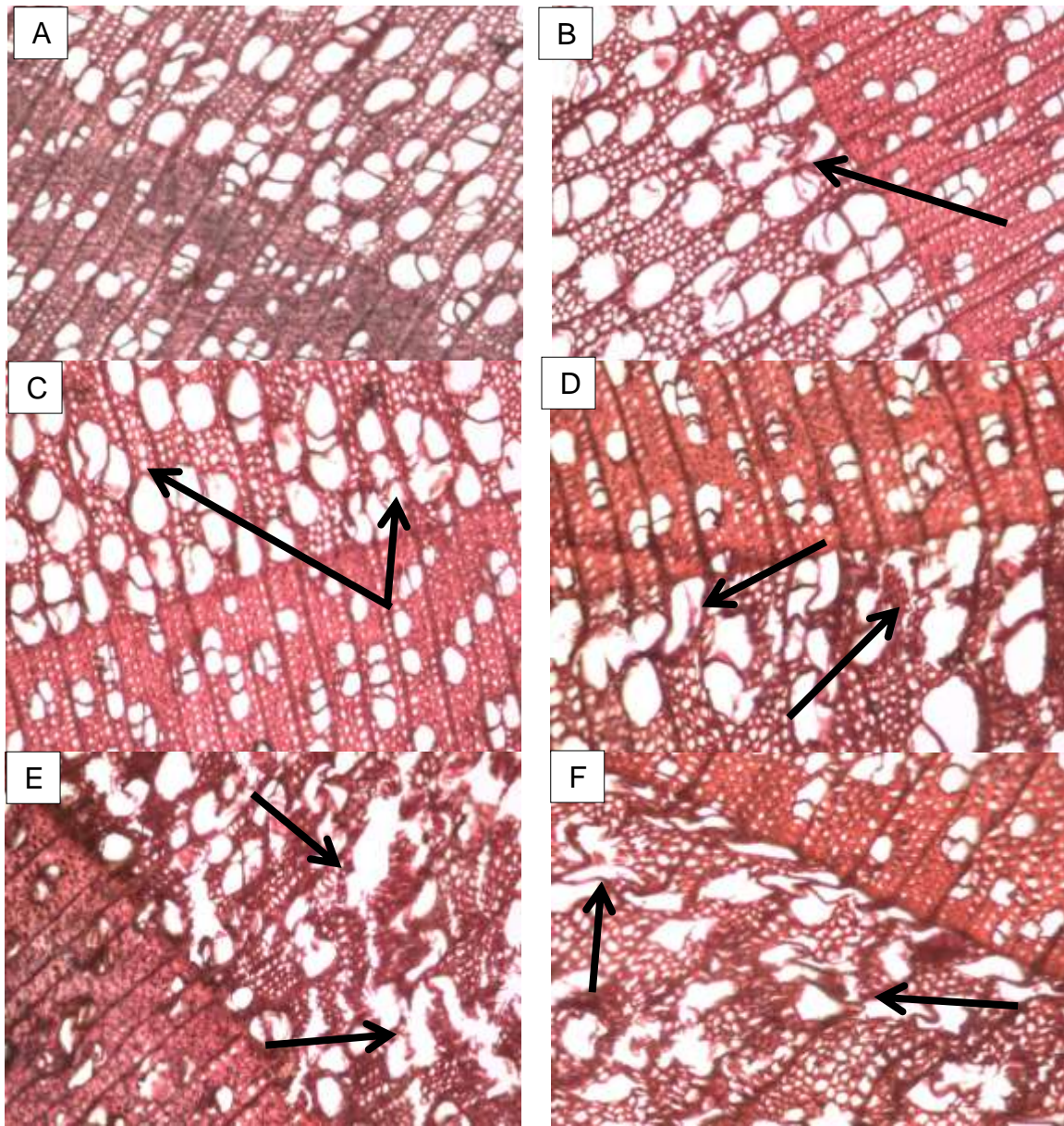


Fig. 1. Images showing collapse of cell structures (B-F) in hot-compressed wood samples: A) control, B) 130 °C, C) 150 °C, D) 170 °C, E) 190 °C, and F) 210 °C

XRD Analysis

Cellulose is comprised of crystal (regular) and amorphous (irregular) zones. Chemical, mechanical, and thermal treatments can affect the crystallinity of the cellulosic materials (Fengel and Wegener 1989). The XRD results of hot-compressed wood samples and control samples are shown in Fig. 2. The crystallinity index (CI) was found by using the wide-angle X-ray diffraction (WAXD) counts at the 2θ angle close to 22° and 18° . The sharp peak at 22° showed the crystalline zone, whereas the peak intensity at 18° reflected to the amorphous zone in cellulosic materials. According to this information, the crystallinity index was calculated using Eq. 2,

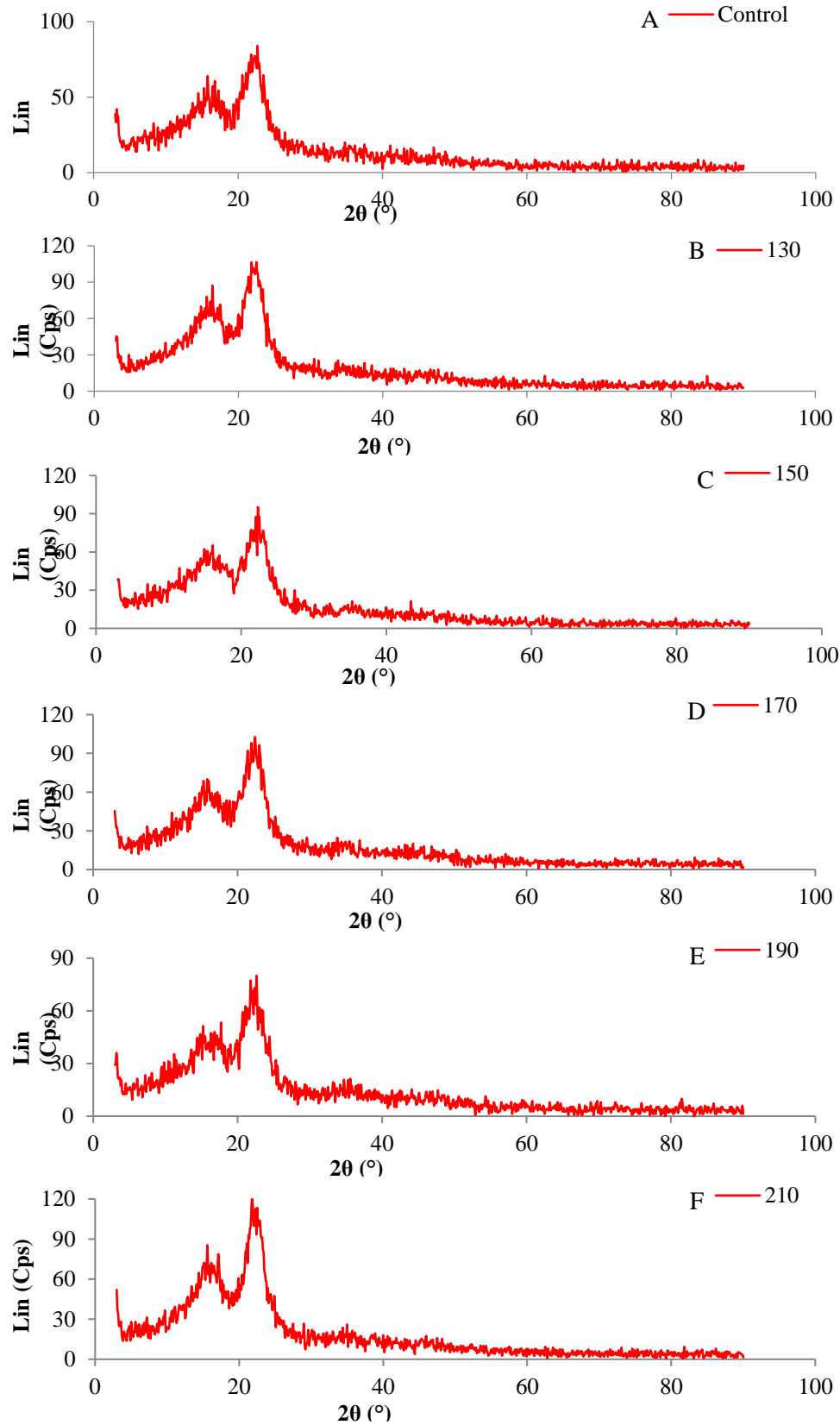


Fig. 2. XRD graphs of hot-compressed wood samples and control sample A) control, B) 130 °C, C) 150 °C, D) 170 °C, E) 190 °C, and F) 210 °C

$$CI (\%) = (I_{22} - I_{18})/I_{22} \quad (2)$$

where I_{22} and I_{18} represent the counter readings at 2θ close to 22° and 18° , respectively (Reddy and Yang 2005). The XRD graphs of all samples are presented in Fig. 2.

The crystallinity index of the control sample and hot-compressed samples are indicated in Table 3.

Table 3. Crystallinity Index of Control Sample and Hot-compressed Samples

Samples	Control	130 °C	150 °C	170 °C	190 °C	210 °C
Crystallinity index (%)	50.5	63.1	63.6	67.5	68.3	68.7

According to Table 3, it was determined that the crystallinity index increased when press temperature increased. The crystallinity index of the control sample was 50.5%, whereas the crystallinity index of the compressed sample at 210 °C rose to 68.7% by increase in a step-by-step fashion.

Colour Change

Colour parameters (L^* , a^* , b^*) and total colour change (ΔE^*) of untreated and heat-treated samples are presented in Table 4. The highest total colour change (39.3) was observed in the heat-treated samples at 210 °C, and the lowest total colour change (5.99) was observed in the heat-treated samples at 130 °C.

Table 4. Colour Change of Heat-treated Wood Samples

Temperature	L^*	a^*	b^*	ΔE
Control	81.55	4.17	21.64	-
130 °C	75.82	5.50	23.14	5.99 (2.31) a
150 °C	74.36	5.88	22.88	7.26 (2.42) a
170 °C	65.88	10.06	24.05	16.68 (2.07) b
190 °C	57.47	10.49	22.14	24.58 (1.56) c
210 °C	42.82	8.80	16.10	39.30 (2.61) d

Note: $p \leq 0.05$, values in parentheses are standard deviations; ^{a,b,c,d,e} values having the same letter are not significantly different (Duncan Test)

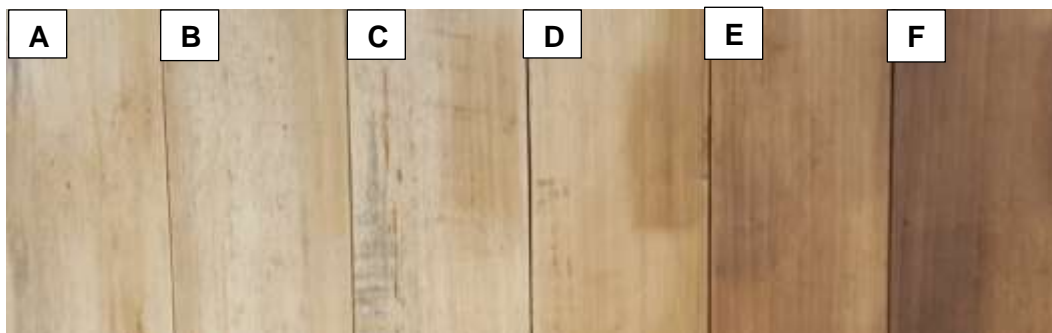


Fig. 3. Darkening poplar wood with hot-pressing: A) control, B) 130 °C, C) 150 °C, D) 170 °C, E) 190 °C, and F) 210 °C

When compared with the control samples, the colour of the heat-treated wood became darker due to the increased temperature. Similar to the findings of this study, several authors stated that as the temperature of the heat treatment increases, the amount of the colour change increases and the wood get a characteristic brown colour (Militz 2002; Esteves and Pereira 2009). Figure 3 shows that the samples became darker with the increase in temperature from 130 °C to 210 °C.

CONCLUSIONS

1. The mechanical characteristics and density of the thermally modified wood were enhanced by this process.
2. As the press temperature increased at constant press pressure, the anatomic deformation, crystallinity index, and colour change of hot-compressed wood increased for high temperatures.
3. Thermal compression treatment was found to be a suitable process for the modification of wood boards. It was thought that the advanced properties of the thermally modified wood boards can bring new approaches to the wood products industry.
4. According to results of this research, hot compression method can used to improve end-use performance of poplar wood which fast growing, low density and relatively low mechanical properties. Moreover, this process makes it possible to evaluate of low density wood species in areas where high mechanical properties are required. It should be improved end-use properties of fast-growing trees for the sustainability of industrial production and sustainable forestry in subsequent research.

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