Effects of Heat Treatment on Some Characteristics of Scots Pine (*Pinus sylvestris* L.) Wood

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Heat treatment of wood materials is generally performed to improve the physical, mechanical, chemical, surface, thermal, and crystallinity characteristics. In this way, the usage areas of wood material in different purposes can be expanded by means of heat treatment. The goal of this study was to determine the physical, mechanical, chemical, crystallinity, and surface properties of heat-treated Scots pine (*Pinus sylvestris* L.) wood. The test samples were heat-treated at 120 °C, 150 °C, 180 °C, and 210 °C for 4 and 6 h in a laboratory-scale oven. The shrinking and swelling chracteristics of wood was decreased as a function of heat treatment processes. Bending strength, compression strength, and modulus of elasticity decreased. In addition, lignin ratios and crystallinity index increased as temperature and duration of the treatment were increased. Consequently, heat-treated wood materials can be used in various areas by developing some of their properties.

Keywords: Thermal treatment; Shrinkage; Swelling; Wood; Crystallinity

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INTRODUCTION

Scots pine (*Pinus sylvestris* L.) belongs to the Pinaceae family and is generally divided into five subspecies. Scots pine (*Pinus sylvestris* L.), which constitutes pure stands or mixed forests with, for example, black pine (*Pinus nigra* J. F. Arnold), spruce (*Picea orientalis* (L.) Link), or oak (*Quercus robur* L.), is one of the most important tree types in Turkey. This tree species expanded from Scotland into Europe, the Alps, the Balkans, Scandinavia, Turkey, Asia, and Siberia. In Turkey, it extends from Yeşildağ, Eskişehir to Oltu, Posof, and Sarıkamış (Anşin and Özkan 2006). Scots pine has a high quality wood that is used in different industries, such as in the production of construction materials in buildings, window frames, furniture, plywood, fiberboard, particle board, and paper (Bozkurt and Erdin 2000).

Heat treatment is one method for improving some mechanical, physical, chemical, thermal, and surface characteristics of wood. Heat-treated wood is preferred to wood treated with environmentally hazardous chemicals (Korkut and Guller 2008; Yildiz *et al*. 2011). Considerable research has focused on the application of heat treatments to improve the dimensional stability, hygroscopic properties, and biological resistance of wood. The heat treatment methods modify the chemical structure of the cell components of the wood. These chemical changes can be expressed as increased dimensional stability and decreased hygroscopicity (Ifju 1964; Yilgor *et al*. 2001; Kartal *et al*. 2008; Kaygin *et al*. 2009). Heat treatment reduces water uptake, and wood cell walls absorb less water because of the decreased amount of hydroxyl groups. In addition to better durability, the advantages of

heat-treated wood include reduced hygroscopicity and improved dimensional stability. After heat treatment process, the moisture content of wood decreased to 4-5%. (Inoue *et al.* 1993). However, some mechanical properties of wood material can decrease after heat treatment (Wikberg and Maunu 2004).

The chemical properties of wood change as a result of heat treatment. Cell wall compounds degrade, and some volatile wood extractives evaporate (Nuopponen *et al*. 2003). Some changes in chemical characteristics improve the dimensional stability of heat-treated wood. These changes are the cross-linking of aromatic rings in lignin, the cross-linking or bridging of cellulose chains as a result of the splitting of two hydroxyl groups on adjacent cellulose chains under high-temperature conditions, and the loss of hygroscopic hemicellulose polymers during heat treatment, which leads to a decrease in hydroxyl groups and consequent reduction of hygroscopicity (Kocaefe *et al*. 2015). Furthermore, the crystallinity properties of heat-treated wood increased compared to untreated samples, and the amorphous regions of the cellulosic structure increased with increasing temperature and time (Akgül *et al.* 2007; Yildiz and Gümüşkaya 2007).

Currently, a number of heat treatment methods are applied successfully at different temperatures and times.These methods have different names, such as the Plato method, the ThermoWood® method, le Bois Perdure, the retification method, and the oil-heat treatment (OHT) method, in the European heat-treated wood market (Sandberg *et al*. 2013).

There are many studies on the effects of heat treatment processes on the physical, mechanical, chemical, and thermal performance of some wood species (de Cademartori *et al*. 2015; Guller 2012; Araújo *et al*. 2016; Percin *et al*. 2016; Icel and Beram 2017). Vỳbohová *et al.* (2018) investigated the effect of heat treatment on chemical composition of ash wood. They concluded that considerable changes in the polysaccharides were caused by the heat treatment of ash wood. The lignin content slightly decreased at the beginning, at a temperature of 160 °C, while at higher temperatures it increased. A detailed discussion on the effects of heat post-treatment on dimensional stability and water absorption behaviours of mechanically densified Uludağ fir and black poplar woods can be found in Pelit *et al.* (2016). They found that, in densified wood samples, heat post-treatment had a significant effect on dimensional stability and water absorptioon. The increrase in temperature of the treatment had a positive effect on obtained results.

The present study aimed to determine the optimum heat treatment conditions, according to wood species. To meet this objective, the present study focused on Scots Pine wood to investigate the physical, mechanical, chemical, surface, and crystallinity properties during the process of heat treatment under various process conditions (temperature and time). The content of the extractives, holocellulose, alpha-cellulose and lignin was determined utilizing the convential methods. To determine the crystallinity indicies (CI), X-ray Diffraction (XRD) was used.

EXPERIMENTAL

Materials

In this study, Scots pine (*Pinus sylvestris* L.) wood specimens were used as test samples, and they were provided from forest product companies in Tosya, Kastamonu, Turkey. Defect-free test samples were sized in dimensions of 20 mm × 20 mm × 30 mm and 20 mm × 20 mm × 300 mm. Heat treatment was performed on the wood samples at 120 °C, 150 °C, 180 °C, and 210 °C for 4 and 6 h in a laboratory-scale oven. The samples

were kept in a conditioning chamber until 12 % moisture content was reached. All experiments were conducted in the labs of Kastamonu University, Department of Forest Industry Engineering and Department of Physics. The control and heat-treated wood samples are shown in Fig. 1.

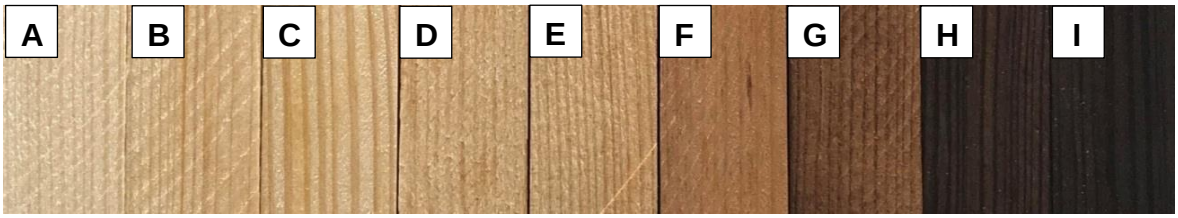

Fig. 1. Heat-treated wood samples: (A) control; (B) 120 °C, 4 h; (C) 120 °C, 6 h; (D) 150 °C, 4 h; (E) 150 °C, 6 h; (F) 180 °C, 4 h; (G) 180 °C, 6 h; (H) 210 °C, 4 h; (I) 210 °C, 6 h

Physical Properties

Test samples measuring 20 mm × 20 mm × 30 mm were used for the determination of shrinkage and swelling ratios, equilibrium moisture content, air-dry density and oven-dry density according to the Turkish standards TS 2472 (1976), TS 4083 (1984), TS 4084 (1984), TS 4085 (1984), and TS 4086 (1984). The specimens were weighed to the nearest 0.01 g and measured to the nearest 0.001 mm immediately. Twelve replicate samples were tested for each treated and untreated group.

Mechanical Properties

The flexural and tensile tests and measurement of compressive strength parallel to the grain were performed according to the Turkish standards TS 2595 (1977), TS 2474 (1977), and TS 2478 (1978), respectively, using a Shimadzu universal testing machine (Shimadzu Corporation, Kyoto, Japan). The tests were performed at a crosshead speed of 5 mm/min. Twelve replicate specimens were tested for the flexural, tensile, and compressive strength parallel to grain measurements.

Chemical Properties

Chemical analyses of the control and heat-treated wood samples were conducted for determination of main wood components according to TAPPI standards. Before the chemical analyses, wood samples were ground in a Wiley mill, and experimental samples were prepared according to the TAPPI T257 cm-85 (1985) standard. Ethanol solubility (TAPPI T204 om-88, 1988), holocellulose (Chlorite method of Wise), alpha-cellulose (TAPPI T203 OS-71, 1999), and lignin ratios (TAPPI T222 om-88, 1988) were obtained as chemical properties of the samples.

Surface Properties

Color measurement and surface roughness

The color changes of untreated and treated wood samples were measured acording to ASTM-D 2244-2 (2011) with a Konica Minolta CM-2500d series spectrophotometer (Tokyo, Japan). The CIELAB color system consists of three parameters (L^*, a^*, and b^*), where L^* is the lightness, and a^* and b^* are the color coordinates. The total color change (ΔE^*) was calculated using the L^*, a^*, and b^* data of each of the untreated and treated samples.

Surface roughness testing was performed according to ISO 4287-1 (1984). Roughness measurements were performed with a portable stylus-contact type roughness meter (Zeiss, Oberkochen, Germany). The pick-up travel length and cut-off length were set to 15 mm and 2.5 mm, respectively. Measurements were performed at a stylus speed of 0.5 mm/s. The equipment was checked every 100 measurements by using a standard reference plate with an average roughness.

X-ray Diffraction (XRD) Analysis

X-ray diffraction analysis was performed with control and heat-treated Scots pine wood samples in dimensions of 20 mm x 20 mm x 30 mm, using a Bruker D8 Advance diffractometer (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° 2θ. The samples were scanned in the range of 5° to 60° 2θ. A step size of 0.02° and a step time of 1.0 s were used during the measurements. A Peltier cooled solid-state [Si(Li)] detector with a useful energy range of 1 keV to 60 keV was used as the detector. No correction was made for Kβ radiation. A set of 2° Soller slits were used to lower the horizontal beam divergence.

Data Analysis

An analysis of variance (ANOVA) was conducted ($p < 0.05$) to evaluate the effects of the temperature and duration on the physical and mechanical characteristics of the Scots pine wood. Significant differences among the average values of the heat-treated woods were determined using Duncan's multiple range tests.

RESULTS AND DISCUSSION

Physical Properties

The equilibrium moisture contents and air- and oven-dry densities of the samples are shown in Table 1.

Table 1. Equilibrium Moisture Contents and Densities of the Heat-treated Wood

Samples		Equilibrium Moisture Content (%)	Air-dry Density (g/cm^3)	Oven-dry Density (g/cm^3)
Control		11.51 (0.003)^{f}	0.528 (0.032)^{nc}	0.501 (0.039)^{nc}
120 °C	4 h	8.89 (0.004)^{e}	0.526 (0.039)^{nc}	0.499 (0.047)^{nc}
	6 h	8.78 (0.003)^{e}	0.525 (0.029)^{nc}	0.495 (0.020)^{nc}
150 °C	4 h	8.23 (0.004)^{d}	0.524 (0.031)^{nc}	0.498 (0.011)^{nc}
	6 h	7.94 (0.005)^{d}	0.523 (0.032)^{nc}	0.494 (0.027)^{nc}
180 °C	4 h	7.42 (0.003)^{c}	0.521 (0.042)^{nc}	0.488 (0.041)^{nc}
	6 h	6.99 (0.003)^{b}	0.514 (0.044)^{nc}	0.488 (0.035)^{nc}
210 °C	4 h	5.75 (0.003)^{a}	0.512 (0.028)^{nc}	0.486 (0.017)^{nc}
	6 h	5.48 (0.002)^{a}	0.505 (0.039)^{nc}	0.478 (0.020)^{nc}

$p \leq 0.05$, values in parentheses are standard deviations; values with the same letter (a, b, c, d, or e) are not significantly different; nc – no correlation (Duncan's test).

The equilibrium moisture content of the heat-treated wood decreased with increasing heat treatment temperature and duration. This result can be attributed to the removal of free and bound water in the wood samples gradually occuring in relation to temperature and duration. Akyildiz and Ateş (2008) reported a decrease of up to 47% in equilibrium moisture content compared to the control samples in their work conducted with oak, chestnut, Turkish red pine, and black pine wood samples. De Cademartori *et al*. (2015) found that equilibrium moisture contents of rose gum (*Eucalyptus grandis* Hill ex Maiden) and Sydney blue gum (*Eucalyptus saligna* Sm.) woods decreased as temperature increased.

The air-dry density of the samples gradually decreased with increasing treatment temperature and duration time. Furthermore, the densities diminished because the weight loss was greater than the volumetric shrinkage as a result of the removal of volatile extractives and moisture in the samples at high temperatures. Decreases in oven-dry density resembled decreases in air-dry density. For the temperatures other than 180 °C, the oven-dry densities of the 6-h heat-treated wood samples were lower than those of the 4-h heat-treated wood samples. Kaygin *et al.* (2009), Korkut and Guller (2008), Gunduz and Aydemir (2009), Gunduz *et al.* (2009), Korkut *et al.* (2008), and Korkut and Bektas (2008) found similar results in their studies. For example, Korkut and Bektas (2008) investigated that the effects of heat treatment on physical properties of Uludag Fir (*Abies bornmuelleriana* Mattf.) and Scots pine (*Pinus sylvestris* L.) wood. They found that heat treatment diminished air-dry and oven-dry density, resulting in higher dimensional stability of heat-treated samples. Shrinkage and swelling ratios of the control and heat-treated samples are shown in Table 2.

Table 2. Shrinkage and Swelling Ratios of the Samples

Samples		Shrinkage (%)	Swelling (%)
Control		11.77 (0.040)^{c}	14.60 (0.017)^{c}
120 °C	4 h	11.70 (0.016)^{bc}	14.27 (0.027)^{c}
	6 h	11.49 (0.016)^{bc}	14.08 (0.023)^{c}
150 °C	4 h	11.41 (0.009)^{bc}	13.90 (0.015)^{c}
	6 h	11.23 (0.015)^{bc}	13.65 (0.013)^{bc}
180 °C	4 h	11.16 (0.012)^{bc}	12.61 (0.031)^{bc}
	6 h	10.58 (0.014)^{abc}	11.81 (0.017)^{b}
210 °C	4 h	9.39 (0.008)^{ab}	9.86 (0.007)^{a}
	6 h	8.89 (0.007)^{a}	9.58 (0.014)^{a}

$p \leq 0.05$, values in parentheses are standard deviations; values with the same letter (a, b, c, d, or e) are not significantly different (Duncan's test).

As shown in Table 2, shrinkage and swelling ratios decreased as temperature and duration increased. The minimum shrinkage and swelling ratios were seen in the 210 °C, 6 h, sample (8.89% and 9.58%, respectively). Korkut and Guller (2008) stated that swelling ratios of red-bud maple (*Acer trautvetteri* Medw.) wood decreased with increasing treatment temperature and duration. In addition, Yang *et al.* (2016) found similar results with Japanese cedar wood. They found that dimensional stability of the Japanese cedar wood improved in response to increased temperatures and prolonged durations. Researchers attributed this to reductions in the hydroxyl groups of wood during heat treatment.

Mechanical Properties

Bending and compression properties and the elastic moduli of the samples were found as mechanical properties. The results of the mechanical tests are shown in Table 3.

Table 3. Results of Mechanical Tests

Samples		Compressive Strength (N/mm^2)	MOR (N/mm^2)	MOE (N/mm^2)
Control		52.59 (4.31)^{bc}	86.41 (11.70)^{d}	9933.03 (1477.43)^{d}
120 °C	4 h	57.52 (5.54)^{bc}	84.16 (13.74)^{cd}	9401.30 (967.88)^{cd}
	6 h	60.31 (4.73)^{bc}	80.08 (8.18)^{cd}	9083.56 (774.43)^{bcd}
150 °C	4 h	57.96 (5.08)^{bc}	80.09 (5.93)^{cd}	8798.48 (374.72)^{bc}
	6 h	57.67 (5.62)^{bc}	78.58 (11.74)^{cd}	8529.20 (501.68)^{b}
180 °C	4 h	56.55 (5.58)^{c}	72.62 (8.01)^{bc}	8511.38 (621.93)^{b}
	6 h	56.08 (3.98)^{c}	70.62 (4.83)^{b}	8463.19 (302.65)^{b}
210 °C	4 h	49.38 (6.73)^{b}	40.87 (8.98)^{a}	7384.81 (741.88)^{a}
	6 h	41.31 (11.29)^{a}	36.08 (10.05)^{a}	7158.78 (888.94)^{a}

$p \leq 0.05$, values in parentheses are standard deviations; values with the same letter (a, b, c, d, or e) are not significantly different (Duncan's test).

As shown in Table 3, compression strength, bending strength, and modulus of elasticity (MOE) decreased with increasing treatment temperature and duration. The lowest compression strength, bending strength, and modulus of elasticity were found in the samples treated at 210 °C for 6 h (41.3, 36.1, and 7160 N/mm^2, respectively). Korkut and Hiziroglu (2009) determined decreases in compression strength, bending strength, and modulus of elasticity (MOE) of heat-treated hazelnut wood (*Corylus colurna* L.) samples as 25.6%, 31.9%, and 27.7% in 180 °C and 10 h treatment, compared to control samples, respectively. Similarly, Candelier *et al*. (2013) found that modulus of rupture (MOR) and modulus of elasticity (MOE) of heat-treated beech woods were decreased by nearly 45% and 12%, respectively. Similar results were reported by Kačíková *et al*. (2013), Zhang *et al*. (2013), Esteves *et al*. (2014), and Kamdem *et al.* (2002). For example Kamdem *et al.* (2002) stated that embrittlement of fibers due to a heat treatment process resulted in loss of strength of wood. Furthermore, wood becomes more brittle due to the degradation of cell wall components and loss of wood mass during heat treatment (Yang *et al.* 2016).

The effects of the heat treatment on compressive strength, MOR and MOE of pine wood are shown in Table 3, associated with the Duncan results. These results indicated that the process temperaures and durations imposes signicant effects on mechanical properties.

Chemical Properties

For determination of chemical properties such as alcohol solubility, holocellulose, alpha cellulose, and lignin content of the heat-treated wood, analyses were performed according to related TAPPI standards. The results obtained are shown in Table 4. As shown, the lowest holocellulose and alpha cellulose ratios as well as the highest lignin ratio were found in samples treated at 210 °C for 6 h. There was not an excessive decrease in the alpha cellulose ratio due to the durable crystalline structure of cellulose. The reduction in the holocellulose ratio of the treated samples was due to degradation of hemicellulose.

The lignin ratio also increased as temperature and treatment time increased. This result confirmed most of the conclusions stated in earlier publications. In other words, it is

evident that the relative increase in lignin content during heat treatment can be attributed to the thermal decomposition products of carbohydrates (Kamdem *et al*. 2002; Kotilainen 2000; Yildiz *et al.* 2006; Nguila Inari *et al.* 2007; Phuong *et al.* 2007).

Table 4. Chemical Properties of the Samples

Samples	Extractives (%)	Holocellulose (%)	Alpha-cellulose (%)	Lignin (%)
Control	6.24	78.45	58.41	20.02
120-4	7.92	77.04	58.68	20.59
120-6	7.98	76.83	58.15	20.60
150-4	10.58	74.16	57.58	20.79
150-6	13.45	73.75	55.15	21.10
180-4	13.34	73.51	54.01	24.40
180-6	9.74	60.07	53.56	30.99
210-4	8.21	54.32	52.18	36.20
210-6	7.19	51.39	50.50	37.50

According to Table 4, the extractives ratio increased then decreased as temperature and duration of heat treatment increased. It is thought that this situation results from evaporation of water in samples followed by evaporation of volatile extractives.

Surface Properties

Surface roughness

Average surface roughness (R_a, R_y, and R_z) values of the Scots pine wood are given in Table 5. When compared with natural control samples and heat-treated samples, minimum reduction of surface roughness occurred with 4-h heat-treated samples at 120 °C, and maximum reduction occurred with 6-h heat-treated samples at 210 °C. Reduction of surface roughness values means that there is also a reduction of roughness on the material surface. Follrich *et al.* (2006) reported that, heat treatment resulted a plastification on the solid wood surfaces. High temperatures above 160 °C cause lignin to a thermoplastic condition and thus to densify and compact solid wood surface. Two-hour heat treatment at 200 °C in Scots pine has been reported to reduce the surface roughness more (Karagoz *et al.* 2011). It has been determined that the surface roughness was reduced in Scots pine with increasing heat treatment period (Ayata *et al.* 2018a). Reductions of surface roughness are associated with degradation of hemicellulose in the cell wall and changes in components (Tomak *et al.* 2014; Ayata *et al.* 2018a).

Table 5. Average Surface Roughness Values

Species	Samples								
	Control	120-4	120-6	150-4	150-6	180-4	180-6	210-4	210-6
R_a (µm)	3.42 (0.48)	2.99 (0.59)	2.91 (0.31)	2.30 (0.32)	2.21 (0.47)	2.19 (0.23)	2.18 (0.67)	2.09 (0.47)	2.07 (0.54)
R_y (µm)	18.71 (2.78)	16.63 (3.69)	16.30 (1.09)	12.60 (2.48)	11.86 (2.71)	11.57 (0.40)	11.77 (0.79)	11.84 (2.13)	10.48 (0.26)
R_z (µm)	12.66 (2.27)	11.32 (2.93)	11.55 (1.45)	8.61 (1.53)	7.48 (1.61)	7.80 (0.27)	7.83 (1.01)	8.12 (1.24)	7.88 (0.75)

Values in parentheses are standard deviations.

Total Color Change

The average total color change values of the Scots pine wood are given in Table 6. Compared with natural control samples and heat-treated samples, the color darkened with increasing heat treatment temperature and duration. Following the heat treatment, the minimum color change according to the natural control samples occurred in 6-h heat-treated samples at 120 °C, and maximum color change occurred in 6-h heat-treated samples at 210 °C. This result shows similarities with those of other studies. Korkut and Kocaefe (2009) reported that the color after heat treatment was darkened due to the oxidative and hydrolytic reactions during the heat treatment. It has been mentioned that changes in wood color after heat treatment are mainly related to degradation of chemical components and removal or decomposition of extracts and other compounds, and the color darkened after heat treatment (Tuncer and Doğu 2018). Atar *et al*. (2019) researched the effect of heat treatment on the color change in varnished wood materials. They found that heat treatment application caused an increase in total color change. A darker color tone occurred in proportion to the increasing temperature and time in the heat treatment (Ayata *et al.* 2018b).

Table 6. Total Color Change Values

	Total Color Change Values (Δ*E**)								
	Control	120-4	120-6	150-4	150-6	180-4	180-6	210-4	210-6
Δ*E**	85.39 (0.79)	82.90 (0.64)	83.21 (1.23)	77.43 (1.65)	74.16 (1.16)	66.69 (1.50)	59.39 (2.96)	33.71 (4.07)	28.24 (6.04)

Values in parentheses are standard deviations

Crystallinity Properties

Chemical, mechanical, and thermal processes can affect the crystallinity properties of cellulose-based materials because the cellulose is composed of crystalline band amorphous zones (Fengel and Wegener 1984). The crystallinity index (CI) was calculated by using wide angle XRD counts at 2θ angle close to 22° and 18°. The sharp peak at 22° demonstrated the presence of crystalline zones, while the peak at 18° corresponded to the amorphous zone in cellulosic materials. Using Eq. 1, the crystallinity index (CI, %) was calculated,

$$CI = (I_{22} - I_{18}) / I_{22} \quad (3)$$

where I_{22} and I_{18} indicate the counter readings at 2θ close to 22° and 18°, respectively (Reddy and Yang 2005). The CI of all samples is shown in Table 7.

Table 7. Crystallinity Indicies of Control and Heat-treated Samples

Samples	Control	120 °C		150 °C		180 °C		210 °C	
		4 h	6 h	4 h	6 h	4 h	6 h	4 h	6 h
CI (%)	70.00	73.68	75.20	76.19	78.72	78.66	80.87	80.53	81.19

According to Table 5, the CI of the heat-treated samples increased gradually, and the maximum CI in the heat-treated samples was observed at 210 °C, 6 h (81.2%). Yildiz and Gümüşkaya (2007) determined that crystallinity properties of cellulose of spruce and beech woods increased with heat treatment. Similarly, Akgül *et al*. (2007) reported that CI of Scots pine (*Pinus sylvestris* L.) and Uludağ fir (*Abies nordmanniana* Stev. subsp. *bornmuelleriana* Mattf.) wood increased as temperature and duration increased. Özgenç *et*

al. (2017) found that high temperature during heat treatment influenced amorphous structures of cellulose, causing an increase in the crystalline part of cellulose. Similarly, Ucuncu *et al*. (2017) stated that CI of wood samples increased when temperature was increased in their study with poplar wood (*Populus* spp.) samples.

CONCLUSIONS

1. Heat treatment affected physical, mechanical, chemical, surface, and crystallinity properties of the Scots pine (*Pinus sylvestris* L.) wood. Equilibrium moisture contents, air-dry densities, oven-dry densities, and shrinkage and swelling ratios of the heat-treated wood samples decreased with increasing treatment temperature and duration, and their minimum values were obtained in samples treated at 210 °C for 6 h.
2. Compression strength, bending strength, and modulus of elasticity (MOE) of the wood decreased with increasing treatment temperature and duration.
3. The surface roughness was reduced in proportion to the increasing heat treatment temperature and duration, while the color darkened, and the hardness was reduced. Especially at temperatures greater than 200 °C, when compared to the control samples, the surface roughness was significantly reduced, the color darkened more, and the hardness was reduced more.
4. The crystallinity index (CI) of the heat-treated samples increased gradually, and the maximum CI in the heat-treated samples was observed in the samples treated at 210 °C for 6 h.
5. The characteristics of the Scots pine wood in this study are of great importance for furniture and chopping industries. Therefore, it is recommended that heat-treated Scots pine wood that is used in furniture, pergolas, and decoration works in areas that are exposed to external influences should be subjected to long-term heat treatment at low temperatures.

REFERENCES CITED

Akgül, M., Gümüşkaya, E., and Korkut, S. (2007). "Crystalline structure of heat-treated Scots pine [*Pinus sylvestris* L.] and Uludağ fir [*Abies nordmanniana* (Stev.) subsp. *bornmuelleriana* (Mattf.)] wood," *Wood Science and Technology* 41, 281-289. DOI: 10.1007/s00226-006-0110-9

Akyildiz, M. H., and Ateş, S. (2008). "Effect of heat treatment on equilibrium moisture content (EMC) of some wood species in Turkey," *Research Journal of Agriculture and Biological Sciences* 4(6), 660-665.

Anşin, R., and Özkan, C. (2006). *Tohumlu Bitkiler (Spermatophyta) Odunsu Taksonlar* [*Seed Plants (Spermatophyta) Woody Taxa*], Karadeniz Technical University Faculty of Forestry Publications, Trabzon, Turkey.

Araújo, S. O., Rocha Vital, B., Oliveira, B., Oliveira Carnerio, A. d. C., Laurenço, A., and Pereira, H. (2016). "Physical and mechanical properties of heat treated wood from *Aspidosperma populifolium*, *Dipteryx odorata* and *Mimosa scabrella*," *Maderas. Ciencia y tecnología* 18(1), 143-156. DOI: 10.4067/S0718-221X2016005000015

ASTM D 2244 (2011). "Standard practice for calculation or color tolerances and color differences from instrumentally measured color coordinates," American Society for Testing and Materials, West Conshohocken, USA.

Atar, M., Yalınkılıç A. C., and Keskin, H., (2019). "Isıl işlemin vernikli ağaç malzemede renk değişimine etkisi [Impact of heat treatment on the exchange of color in varnished wood materials]," *Journal of Polytechnic* 22(2), 407-413. DOI: 10.2339/politeknik.404008

Ayata, Ü., Gürleyen, T., and Gürleyen, L. (2018b). "Effect of heat treatment on colour and glossiness properties of zebrano, sapelli and merbau woods," *Furniture and Wooden Material Research Journal* 1(1), 11-20. DOI: 10.33725/mamad.428913

Ayata, Ü., Gürleyen, T., Gürleyen, L., and Çakıcıer, N. (2018a). "Determination of surface roughness parameters of heat-treated and untreated Scotch pine, oak and beech woods," *Furniture and Wooden Material Research Journal* 1(1), 46-50. DOI: 10.33725/mamad.433945

Bozkurt, A. Y., and Erdin, N. (2000). *Odun Anatomisi*, Istanbul University Faculty of Forestry Publications, Faculty Issue: 466, İstanbul, Turkey.

Candelier, K., Dumarçay, S., Pétrissans, A., Gérardin, P., and Pétrissans, M. (2013). "Comparison of mechanical properties of heat treated beech wood cured under nitrogen or vacuum," *Polymer Degradation and Stability* 98(9), 1762-1765. DOI: 10.1016/j.polymdegradstab.2013.05.026

de Cademartori, P. H. G., Missio, A. L., Mattos, B. D., and Gatto, D. A. (2015). "Effect of thermal treatments on technological properties of wood from two *Eucalyptus* species," *Anais da Academia Brasileira de Ciências* 87(1), 471-481. DOI: 10.1590/0001-3765201520130121

Esteves, B., Nunes, L., Domingos, I., and Pereira, H. (2014). "Comparison between heat treated sapwood and heartwood from *Pinus pinaster*," *European Journal of Wood and Wood Products* 72(1), 53-60. DOI: 10.1007/s00107-013-0751-y

Fengel, D., and Wegener, G. (1984). *Wood—Chemistry, Ultrastructure, Reactions*, Walter de Gruyter, New York, NY, USA. DOI: 10.1002/pol.1985.130231112

Follrich, J., Muller, U., and Gindl, W. (2006). "Effects of thermal modification on the adhesion between spruce wood (*Picea abies* Karst.) and a thermoplastic polymer," *Holz Roh-Werkst* 64, 373-376.

Guller, B. (2012). "Effects of heat treatment on density, dimensional stability and color of *Pinus nigra* wood," *African Journal of Biotechnology* 11(9), 2204-2209. DOI: 10.5897/AJB11.3052

Gunduz, G., and Aydemir, D. (2009). "Some physical properties of heat-treated hornbeam (*Carpinus betulus* L.) wood," *Drying Technology* 27(5), 714-720. DOI: 10.1080/07373930902827700

Gunduz, G., Aydemir, D., and Karakas, G. (2009). "The effects of thermal treatment on the mechanical properties of wild Pear (*Pyrus elaeagnifolia* Pall.) wood and changes in physical properties," *Materials & Design* 30(10), 4391-4395. DOI: 10.1016/j.matdes.2009.04.005

Icel, B., and Beram, A. (2017). "Effects of industrial heat treatment on some physical and mechanical properties of iroko wood," *Drvna Industrija* 68(5), 229-239. DOI: 10.5552/drind.2017.1720

Ifju, G. (1964). "Tensile strength behavior as a function of cellulose in wood," *Forest Products Journal* 14(8), 366-372.

Inoue, M., Norimoto, M., Tanahashi, M., and Rowell, R. M. (1993). "Steam or heat fixation of compressed wood," *Wood and Fiber Science* 25(3), 224-235.

ISO 4287-1 (1984). "Surface roughness – Terminology – Part 1: Surface and its parameters," International Organization for Standardization, Geneva, Switzerland.

Kačíková, D., Kačík, F., Čabalová, I., and Ďurkovič, J. (2013). "Effects of thermal treatment on chemical, mechanical and colour traits in Norway spruce wood," *Bioresource Technology* 144, 669-674. DOI: 10.1016/j.biortech.2013.06.110

Karagoz, U., Akyildiz, M. H., and Isleyen, O. (2011). "Effect of heat treatment on surface roughness of thermal wood machined by CNC," *Pro Ligno* 7(4), 50-58.

Kartal, S. N., Hwang, W.-J., and Imamura, Y. (2008). "Combined effect of boron compounds and heat treatments on wood properties: Chemical and strength properties of wood," *Journal of Materials Processing Technology* 198(1-3), 234-240. DOI: 10.1016/j.jmatprotec.2007.07.001

Kaygin, B., Gunduz, G., and Aydemir, D. (2009). "Some physical properties of heat-treated paulownia (*Paulownia elongata*) wood," *Drying Technology* 27(1), 89-93. DOI: 10.1080/07373930802565921

Kocaefe, D., Huang, X., and Kocaefe, Y. (2015). "Dimensional stabilization of wood," *Current Forestry Reports* 1(3),151-161. DOI: 10.1007/s40725-015-0017-5

Korkut, D. S., and Guller, B. (2008). "The effects of heat treatment on physical properties and surface roughness of red-bud maple (*Acer trautvetteri* Medw.) wood," *Bioresource Technology* 99(8), 2846-2851. DOI: 10.1016/j.biortech.2007.06.043

Korkut, D. S., Korkut, S., Bekar, I., Budakçi, M., Dilik, T., and Çakicier, N. (2008). "The effects of heat treatment on the physical properties and surface roughness of Turkish hazel (*Corylus colurna* L.) Wood," *International Journal of Molecular Sciences* 9(9), 1772-1783.

Korkut, S., and Hiziroglu, S. (2009). "Effect of heat treatment on mechanical properties of hazelnut wood (*Corylus colurna* L.)," *Materials and Design* 30,1853-1858.

Korkut, S., and Kocaefe, D. (2009). "Effect of heat treatment on wood properties," *Düzce University Journal of Forestry* 5(2), 11-34.

Korkut, S., and Bektas, I. (2008). "The effects of heat treatment on physical properties of Uludag fir (*Abies bornmuelleriana* Mattf.) and Scots pine (*Pinus sylvestris* L.) wood," *Forest Product Journal* 58(3), 95-99.

Kotilainen, R. (2000). *Chemical Changes in Wood during Heating at 150-260°C*, Ph.D. Dissertation, Jyväskylä University, Jyväskylä, Finland.

Nguila Inari, G., Petrissans, M., and Gerardin, P. (2007). "Chemical reactivity of heat-treated wood," *Wood Science and Technology* 41(2), 157-168. DOI: 10.1007/s00226-006-0092-7

Nuopponen, M., Vuorinen, T., Jämsä, S., and Viitaniemi, P. (2003). "The effects of a heat treatment on the behavior of extractives in softwood studied by FTIR spectroscopic methods," *Wood Science and Technology* 37, 109-115.

Özgenç, Ö., Durmaz, S., Boyaci, I. H., and Eksi-Kocak, H. (2017). "Determination of chemical changes in heat-treated wood using ATR-FTIR and FT Raman spectrometry," *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy* 171, 395-400. DOI: 10.1016/j.saa.2016.08.026

Pelit, H., Budakcı, M., and Sonmez, A. (2016). "Effects of heat post-treatment on dimensional stability and water absorption behaviours of mechanically densified Uludağ fir and black poplar woods," *BioResources* 11(2), 3215-3229.

Percin, O., Peker, H., and Atilgan, A. (2016). "The effect of heat treatment on the some physical and mechanical properties of beech (*Fagus orientalis* Lipsky) wood," *Wood Research* 61(3), 443-456.

Phuong, L. X., Shida, S., and Saito, Y. (2007). "Effects of heat treatment on brittleness of *Styrax tonkinensis* wood," *Journal of Wood Science* 53, 181-186. DOI: 10.1007/s10086-006-0841-0

Reddy, N., and Yang, Y. (2005). "Structure and properties of high quality natural cellulose fibers from cornstalks," *Polymer* 46(15), 5494-5500. DOI: 10.1016/j.polymer.2005.04.073

Sandberg, D., Haller, P., and Navi, P. (2013). "Thermo-hydro and thermo-hydro-mechanical wood processing : An opportunity for future environmentally friendly wood products," *Wood Material Science & Engineering* 8(1), 64-88. DOI: 10.1080/17480272.2012.751935

TAPPI T203 OS-71 (1999). "Alpha-, beta-, and gamma-cellulose in pulp," TAPPI Press, Atlanta, GA.

TAPPI T204 om-88 (1988). "Wood extractives in ethanol-benzene mixture," TAPPI Press, Atlanta, GA.

TAPPI T222 om-88 (1988). "Acid-insoluble lignin in wood and pulp," TAPPI Press, Atlanta, GA.

TAPPI T257 cm-85 (1985). "Sampling and preparing wood for analysis," TAPPI Press, Atlanta, GA.

Tomak, E. D., Ustaomer, D., Yildiz, S., and Pesman, E. (2014). "Changes in surface and mechanical properties of heat treated wood during natural weathering," *Measurement* 53, 30-39. DOI: 10.1016/j.measurement.2014.03.018

Tuncer, F. D., and Doğu A. D. (2018). "Effects of heat treatment on some macroscopic and physical properties of Scots pine sapwood and heartwood," *Forestist* 68(2), 93-100. DOI: 10.26650/forestist.2018.343295

TS 2472 (1976). "Wood - Determination of density for physical and mechanical tests," Turkish Standards Institute, Ankara, Turkey.

TS 2474 (1977). "Determination of static bending strength of wood," Turkish Standards Institute, Ankara, Turkey.

TS 2478 (1978). "Wood determination of static modulus of elasticity in static bending," Turkish Standards Institute, Ankara, Turkey.

TS 2595 (1977). "Determination of pressure strength in parallel to wood fibers," Turkish Standards Institute, Ankara, Turkey.

TS 4083 (1984). "Wood - Determination of radial and tangential shrinkage," Turkish Standards Institute, Ankara, Turkey.

TS 4084 (1984). "Wood - Determination of radial and tangential swelling," Turkish Standards Institute, Ankara, Turkey.

TS 4085 (1984). "Wood - Determination of volumetric shrinkage," Turkish Standards Institute, Ankara, Turkey.

TS 4086 (1984). "Wood - Determination of volumetric swelling," Turkish Standards Institute, Ankara, Turkey.

Ucuncu, T., Durmaz, E., and Kaymakci, A. (2017). "Characteristics of hot-compressed poplar wood boards," *BioResources* 12(4), 9038-9049. DOI: 10.15376/biores.12.4.9038-9049

Vỳbohová, E., Kućerová, V., Andor, T., Baláźová, Ž., and Vel'ková, V. (2018). "The effect of heat treatment on the chemical composition of ash wood," *BioResources* 13(4), 8394-8408.

Wikberg, H., and Maunu, S. L. (2004). "Characterization of thermally modified hard- and softwoods by ^{13}C CPMAS NMR," *Carbohydrate Polymers* 58(4), 461-466. DOI: 10.1016/j.carbpol.2004.08.008

Yildiz, S., and Gümüşkaya, E. (2007). "The effects of thermal modification on crystalline structure of cellulose in soft and hardwood," *Building and Environment* 42(1), 62-67. DOI: 10.1016/j.buildenv.2005.07.009

Yildiz, S., Gezer, E. D., and Yildiz, U. C. (2006). "Mechanical and chemical behavior of spruce wood modified by heat," *Building and Environment* 41(12), 1762-1766. DOI: 10.1016/j.buildenv.2005.07.017

Yildiz, S., Yildiz, U. C., and Tomak, E. D. (2011). "The effects of natural weathering on the properties of heat-treated alder wood," *BioResources* 6(3), 2504-2521.

Yilgor, N., Unsal, O., and Kartal, S. N. (2001). "Physical, mechanical, and chemical properties of steamed beech wood," *Forest Products Journal* 51(11), 89-93.

Zhang, Y. M., Yu, Y. L., and Yu, W. J. (2013). "Effect of thermal treatment on the physical and mechanical properties of *phyllostachys pubescen* bamboo," *European Journal of Wood and Wood Products* 71(1), 61-67. DOI: 10.1007/s00107-012-0643-6

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