Some of the Properties of Heat-Treated Sessile Oak (Quercus petraea)

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Abstract

The objective of this study was to investigate the effects of heat treatment on the physical and mechanical properties of sessile oak (Quercus petraea). Samples were exposed to three temperature levels of 120° C, 150° C, and 180° C for time periods ranging from 2 to 10 hours. Modulus of elasticity, modulus of rupture, compression strength parallel-to-grain, hardness, impact bending, tension strength, swelling in three sections, and surface roughness of the samples were evaluated. Based on the findings in this study, the results showed a significant difference between properties of control samples and heat-treated samples ($P = 0.05$). Mechanical properties of the samples were adversely influenced as a result of heat treatment; however, surface quality and dimensional stability of the samples improved with heat treatment. Changes in properties of the samples were more pronounced as temperature and exposure times were increased.

 $\mathbf I$ he genus *Quercus* is native to the northern hemisphere, extending from temperate areas to tropical Asia and North America. Sessile oak (Quercus petraea) is also known as Durmast oak and is a native species in most European countries and Anatolia (Yaltirik and Efe 2000). It spreads over 6,068,920 ha, which is almost 29 percent of the total forest land of Turkey (Konukcu 1998, Anonymous 2001).

Sessile oak trees may be up to 30 m in height and have excellent mechanical properties. The species is important in the lumber industry in Turkey and is widely used for different applications including manufacturing of furniture, axles, handles, splitting wedges, novelty items, interior paneling, flooring, timber frame, veneer, wine barrels, fuel, and charcoal (Bozkurt and Erdin 1997, 1998). It is well known that solid wood has certain undesirable properties such as poor resistance against biological deterioration and dimensional instability due to its hygroscopic nature (Kocaefe et al. 2008b).

Based on past studies, heat treatment at relatively high temperatures, ranging from 150° C to 260° C, was found to be an effective method to improve dimensional stability and durability of wood (Seborg et al. 1953; Kollmann and Schneider 1963; Stamm 1964; Kollmann and Fengel 1965; Noack 1969; Burmester 1973, 1975; Giebeler 1983; Hillis 1984; Bourgois and Guyonnet 1988; Sanli 2008). Thermally treated wood has been investigated since the middle of the last century, and thermal modification of wood has become a well-established procedure. There is a growing number of industrial treatment centers in various European countries.

Thermally treated products manufactured by thermowood (Stellac) in Finland, torrefaction (perdure) in France, and PLATO-wood in the Netherlands are some of the examples of this technology (Inoue et al. 1993, Boonstra et al. 1998, Militz 2002, Anonymous 2003, Kocaefe et al. 2008b). Temperatures over 150° C permanently alter the physical and chemical properties of wood. Having higher treatment temperature also enhances biological durability, but some undesirable effects such as reduction of strength and hardness of wood are inevitable (Sandermann and Augustin 1963, Rusche 1973, Kubojima et al. 2000). Thermally treated wood is more brittle, and bending and tension strength characteristics are decreased by 10 to 30 percent. Therefore, the use of heat-treated wood in load-bearing applications is not suggested (Anonymous 2003).

The change in properties of wood is mainly caused by thermic degrading of hemicelluloses. Theoretically, the available hydroxyl groups in hemicellulose have the most significant effect on the physical properties of wood. The

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^a Number of samples in each test was 30. Avg = average; $CV =$ coefficient of variation. Within each column, values with the same letters are not significantly different between samples according to Duncan's multiple range test at $P < 0.05$.

process of heat treatment is generally accompanied by breakage of the lignin-polysaccharide complex by organic acids released from hemicelluloses (Kocaefe et al. 2008a). The treatment reduces water intake and the wood cell wall absorbs less water because of the decrease of the amount of hydroxyl groups in the cell wall. As a result of the reduced number of hydroxyl groups, swelling and shrinking of wood are reduced. Additionally heat treatment results in varying amounts of weight loss, depending on the treatment temperature and exposure time. In a previous study, spruce (Picea abies) wood heat treated for 24 hours showed a weight loss of 0.8 and 15.5 percent at 120° C and 200° C, respectively (Fengel 1966). Weight loss in beech (Fagus sylvatica) wood, treated at increased temperatures was found in another study as 8.1 and 9.8 percent at 150° C and 200°C, respectively (Fengel and Wegener 1989). Red-brown discoloration of wood is also an important characteristic of heat-treated material (Bekhta and Niemz 2003). Such wood is suggested as an alternative material to dark-colored wood products from tropical species (Bourgois et al. 1991, Syrjänen et al. 2000, Bekhta and Niemz 2003). In general the change in timber properties during heat treatment depends mainly on temperature, temperature gradient,

amount of water vapor, and heating time (Zaman et al. 2000).

The improved characteristics of heat-treated sessile oak wood could offer various potential opportunities to the timber product industry. Heat-treated wood is also an ecofriendly alternative to chemically treated wood, having uses for kitchen and sauna furniture, bathroom cabinets, and flooring material (Syrjanen and Oy 2001). To our knowledge, there is no information about the influence of heat treatment on the mechanical and physical properties of sessile oak grown in Turkey. Therefore, the main objective of this study was to evaluate the effect of heat treatment on the properties of sessile oak and to provide preliminary data so that treated wood can be used more widely and effectively by the wood product industry.

Materials and Methods

Five trees with an average age of 95 years and an average diameter at breast height of 38 cm were harvested at a site with an altitude of 850 m and a slope of 60 percent in northeastern Turkey (Turkish Standard [TS] 1984). Lumbers from the logs were sawn and planned (TS 1976a, TS 1981). Following air drying of the material, small and clear

474 KORKUT ET AL.

Table 2.—Reduction in physical properties of the samples as function of heat treatment.

Heat treatment and time	Ovendry density $(\%)$	Air-dry density $(\%)$	Swelling $(\%)$			Surface roughness $(\%)$			
			Radial	Tangential	Longitudinal	R_a	R_{v}	R_z	R_q
120° C									
2 _h	0.918	1.272	3.64	5.527	9.062	6.42	1.36	3.268	0.73
6 h	1.085	2.707	4.677	5.559	10.17	7.05	5.67	4.257	5.77
10 _h	1.376	3.193	10.61	12.08	19.4	7.26	6.62	5.77	6.53
150° C									
2 _h	2.844	3.215	11.02	14.89	21.31	9.73	6.88	6.09	7.64
6 h	3.715	3.908	15.47	15.57	23.83	11.85	9.25	8.824	8.35
10 _h	7.788	4.231	16.97	22.67	24.62	12.81	9.81	10.44	8.98
180° C									
2 _h	7.87	4.416	18.65	26.22	27.95	13	12.4	11.96	13.5
6 h	8.178	8.481	24.56	28.23	28.83	13.82	13.6	17.45	14.3
10 _h	11.2	11.11	31.59	35.73	31.93	25.67	31.5	38.33	24.1

specimens were cut from the boards according to TS 2472, which is similar to ISO 3131 Standard to determine air-dry and ovendry densities $(D_{m12}, D_{m0};$ TS 1976b, International Organization for Standardization [ISO] 1975), swelling (tangential, radial, longitudinal $[\alpha(t, r, 1)]$; TS 1983), compression strength parallel to grain ($\sigma_{c/l}$; TS 1976c), modulus of rupture (MOR; TS 1976d), modulus of elasticity in bending (MOE; TS 1976e), Janka hardness (crosssection, radial and tangential grain orientation [Hj]; TS 1982), impact bending strength (σ_i ; TS 1976f), and tension strength perpendicular to grain ($\sigma_{z\perp}$; TS 1976g). Specimens were divided into nine treatment groups, and a total of 30 test and 30 control samples were used from each group. The samples were subjected to heat treatment at 120° C, 150° C, and 180° C for 2, 6, and 10 hours in a laboratory-type heating unit controlled to an accuracy of $\pm 1^{\circ}$ C of temperature under the atmospheric pressure. Then the specimens were

conditioned at a temperature of 20 \pm 2°C with 65 percent relative humidity according to TS 642 (TS 1997). The moisture content of the samples was 0 percent after heat treatment. Following heat treatment, samples reached to equilibrium moisture content ranging from 9 to 11 percent in a conditioning room. The air-dry density of the samples was also determined. Oven-dried specimens were measured and weighted at an accuracy of 0.01 mm and 0.001 g to determine their density. In the following step the samples were soaked in water at a temperature of $20 \pm 2^{\circ}$ C for 1 week to determine their swelling. Moisture content (M) of each specimen was also measured according to TS 2471 (TS 1976h), and strength values were corrected based on 12 percent moisture content by using the following strength conversion equation:

$$
\delta_{12} = \delta_m[1 + \alpha(M_2 - 12)]
$$

Figure 1.—Reduction in physical properties of the samples as function of heat treatment. (\diamond) Ovendry density; (\square) air-dry density; (\triangle) radial swelling; (\times) tangential swelling; (\divideontimes) longitudinal swelling; (\circ) surface roughness (R_a).

^a The number of samples in each test was 30. Avg = average; $CV =$ coefficient of variation. Within each column, values with the same letters are not statistically different between samples according to the Duncan's multiple range test at $P < 0.05$. Comparisons were between each control and its test.

where

 δ_{12} = strength at 12 percent moisture content (N/mm²),

- $\delta_{\rm m}$ = strength at moisture content deviated from 12 percent (N/mm²),
- α = constant value showing relationship between strength and moisture content ($\alpha = 0.05, 0.04,$ 0.02, 0.025, 0.015, and 0.025 for σ _c//, MOR, MOE, σ_i , $\sigma_{z\perp}$, and Hj, respectively), and
- M_2 = moisture content during test (%).

Surface roughness of the samples was measured by using a stylus-type profilometer (Mitutoyo Surftest SJ-301). The tracing speed, stylus tip diameter, and tip angle were 10 mm/min, 4 μ m, and 90°, respectively. Roughness measurements were taken randomly from the surface of the samples perpendicular to the grain orientation. Three roughness parameters, mean arithmetic deviation of profile (R_a) , mean peak-to-valley height (R_z) , maximum roughness (R_y) , and root mean square (R_q) , which were commonly used in previous studies, were used to evaluate surface characteristics of the samples (Stombo 1963). R_a is the average distance from the profile to the mean line over the length of assessment. R_z can be calculated from the peak-to-valley values of five equal lengths within the profile, while maximum roughness (R_v) is the distance between peak and valley points of the profile that can be used as an indicator of the maximum defect height within the assessed profile (Hiziroglu 1996). Detailed specifications of these parameters are described in past studies (Mummery 1993, Hiziroglu 1996, ISO 1997). Roughness values were measured with a sensitivity of $0.5 \mu m$. The tracing length (Lt) was 15 mm and the cut-off length was $\lambda = 2.5$ mm. The measuring force of the stylus on the surfaces was 4 mN (0.4 g), which did not put any significant damage on the surface. For all parameters, multiple comparisons were first subjected to an analysis of variance (ANOVA) and significant differences between average values of control and treated samples were determined using Duncan's multiple range test at P value of 0.05 (Kalipsiz 1994).

Results and Discussion

Table 1 displays densities, swelling ratios, and surface roughness parameters of the samples exposed to different treatments. In general ovendry and air-dry density values of the samples decreased insignificantly with increasing

Figure 2.—Reduction in mechanical properties of the samples as function of heat treatment. (\diamond) Compression strength; (*) modulus of rupture; (\triangle) modulus of elasticity; (\times) Janka hardness cross section; (\bigcirc) Janka hardness radial; (\blacktriangle) Janka hardness tangential; $(+)$ impact bending strength; $(-)$ tension strength perpendicular to grain.

temperature and treatment periods. Samples exposed to a temperature of 180° C for 10 hours resulted in the lowest airdry and ovendry density values as compared with those treated under other conditions. Reduction in densities, and radial, tangential, and longitudinal swelling values of the samples exposed to a temperature of 180° C for 10 hours were 11.20, 11.11, 31.59, 35.73, and 31.77 percent, respectively. Table 2 and Figure 1 also present overall reduction of physical properties of the samples as a result of heat treatment. Low swelling value of the samples is always desirable because it results in better dimensional stability, which can be related to material losses in the cell wall, extractive substances, and degradation of hemicellulose due to the effect of high temperature. It is well known that the weight of wood and its swelling decrease when heat treatment is applied. Heat treatment reduces the hydrophilic behavior of the wood by modifying the chemical structure of some of its components (Raimo et al. 1996, Gailliot 1998, Homan et al. 2000). Such modification prevents the reabsorption of water that would promote wood decay. When wood absorbs moisture from its surroundings, water molecules are attached between and within the wood polymers, lignin, cellulose, and hemicelluloses, so that hydrogen bonds are formed. This phenomenon causes swelling of wood. During heat treatment, the number of hydrophilic OH groups is decreased and they are replaced by hydrophobic O-acetyl groups (Esteves et al. 2007). This creates cross-links between wood fibers and thus it significantly reduces the ability of the water to penetrate into the wood (Homan et al. 2000). Heat-treated wood becomes dimensionally more stable compared to untreated wood. Elimination of hydroxyl groups also reduces the number of potential anchor-points for fungi (Poncsák et al. 2006).

Water absorption of the cell wall because of the decrease in the amount of wood's hydroxyl groups is enhanced. Consequently, having reduced number of hydroxyl groups improves swelling and shrinking of wood (Follrich et al. 2006, Yildiz et al. 2006).

Surface roughness of the samples treated at a temperature of 180°C for 10 hours had improved R_a values by up to 25.67 percent, which was lower than those of control samples. The heat treatment resulted in a plastification on the solid wood surfaces. High temperatures above 160° C cause conversion of lignin into a thermoplastic condition, which increases density and compacting of the surface layer. As a result, in our study surface characteristics of the samples were noticeably enhanced. Solid wood with a rough surface requires more sanding, which not only wastes raw material by decreasing its thickness but also increases overall production cost. Therefore, it appears that heat treatment would be a potential method to improve surface quality of sessile oak.

Table 3 shows the results of mechanical properties of the samples as a result of heat treatment. All mechanical properties of the specimens were adversely affected by the heat treatment. It is known that equilibrium moisture content of heat-treated wood is generally lower than that of regular wood. However, modification in chemical structure of wood is more responsible for reduced strength properties of the samples exposed to heat treatment. The differences between strength properties of the treated and control samples were statistically significant. The samples treated at a temperature of 180° C for 10 hours resulted in a maximum reduction in all mechanical properties. The lowest compression strength value was 50.42 N/mm², and the total loss compared with the control samples was 21.30 percent. Similarly, the lowest MOR value was 85.52 N/mm² for the samples treated at the above conditions (Sanli 2008).

Table 4.—Reduction in mechanical properties of the samples as function of heat treatment.

Heat treatment and time	Compression strength $(\%)$	Bending strength $(\%)$	Modulus of elasticity in bending $(\%)$	Janka hardness $(\%)$				Tension strength
				Cross section	Radial	Tangential	Impact bending strength $(\%)$	perpendicular to grain $(\%)$
120° C								
2 _h	1.459	5.158	15.868	3.871	1.585	2.374	12.468	3.657
6 h	4.368	7.253	19.584	4.504	2.378	7.035	18.621	6.678
10 _h	4.460	8.848	19.826	4.694	5.037	10.130	18.745	7.693
150° C								
2 _h	5.701	12.943	36.302	5.327	7.001	10.273	21.462	8.275
6 h	6.573	13.117	37.233	6.488	9.166	11.705	23.756	8.773
10 _h	7.276	19.528	37.604	7.453	9.398	11.891	25.248	14.027
180° C								
2 _h	11.597	20.460	39.758	10.236	10.858	13.990	28.699	35.195
6 h	11.730	23.279	42.415	13.333	13.749	16.774	39.276	40.234
10 _h	21.309	35.317	45.585	17.419	17.665	19.016	45.742	45.850

Corresponding average values for MOE, impact strength, tensile strength perpendicular-to-grain, hardness for cross section, and radial and tangential directions were 7,553 N/ mm², 4.428 J/cm², 3.138 N/mm², 57.87 N/mm², 28.38 N/ mm², and 30.35 N/mm², respectively. Figure 2 and Table 4 show reduction in mechanical properties of the samples due to heat treatment. The compression strength and modulus of elasticity of the samples were greatly affected by initial heat treatment, but after that they showed a gradual decrease in their properties with increasing temperatures and treatment times. In general the results of this study in terms of the effect of heat treatment on properties of the samples are comparable to those of a previous study using different species including *Pinus pinaster* and *Eucalyptus globulus* (Esteves et al. 2007). Samples of these two species treated by a temperature ranging between 190° C and 210° C for 2 to 12 hours resulted in increased dimensional stability and surface wettability. Mass losses of the samples increased with increasing treatment time and temperature, reaching 7.3 percent for pine and 14.5 percent for eucalyptus samples. In another study, heat-treated Turkish river red gum (Eucalyptus camaldulensis Dehn.) samples showed reductions in hardness of cross-section, radial, and tangential sections for 23.91, 44.20, and 33.57 percent, respectively, due to similar heat treatment conditions used in this work (Unsal et al. 2003, Unsal and Ayrilmis 2005).

Compression strength parallel-to-grain of Uludag fir (Abies bornmuellerinana Mattf.) samples decreased 29.41 percent when they were exposed to a temperature of 180° C for 10 hours (Korkut 2008). In the same study, 60.56 N/mm² was the lowest MOR value with a reduction of 29.28 percent as compared with control samples. Effect of heat treatment on mechanical properties of the samples from Camiyanı black pine (Pinus nigra Arn. subsp. pallasiana var. pallasiana) and red-bud maple (Acer trautvetteri Medw.) were also investigated and reduction in different strength properties of the samples were found to be similar to those found in this study (Gunduz et al. 2008, Korkut et al. 2008).

In general the decreases in the strength properties of heat-treated sessile oak samples can be related to thermal degradation and lost of chemical building elements of wood to a certain extent as a result of heat treatment. Strength reduction of the samples is mainly due to the depolymerization reactions of wood polymers (Ayrilmis and Winandy 2009). Degradation of hemicelluloses, which is less resistant to heat than cellulose and lignin, could also be one of the main reasons. Changes in and loss of hemicelluloses play key roles in the strength properties of wood heated at high temperatures (Kamden et al. 1999, Kotilainen 2000).

Conclusions

This study demonstrated that all mechanical properties of the heat-treated sessile oak samples tested in this work had some reduction as compared with control samples. However, it appears that heat treatment had a limited benefit on dimensional stability and surface roughness of the samples. Treatment temperature was not the only parameter contributing to the reduction of mechanical properties; weight loss directly connected to treatment duration. Due to reduced mechanical properties of sessile oak as a result of heat treatment, it is recommended that such units should not be used for constructional applications.

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