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Full Length Research Paper

Effects of heat treatment on density, dimensional stability and color of *Pinus nigra* wood

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The purpose of this study was to evaluate the effect of heat treatment on some physical properties and color change of *Pinus nigra* wood which has high industrial use potential and large growing stocks in Turkey. Wood samples which comprised the material of the study were obtained from an industrial plant. Samples were subjected to heat treatment process of varying temperatures (190, 200, 212 and 225°C) and durations (60, 120 and 180 min). After these processes, density and swelling of the wood samples were tested in comparison with untreated ones. The results show that density decreased (2.57 to 12.6%) as treatment temperature and duration increased. Dimensional stability was improved (up to 66%) with the extent of the circumstances mainly depending on treatment temperature. Color became uniformly darker with increasing temperature and durations. Temperature had clearly greater influence on investigated properties than duration.

Key words: *Pinus nigra*, heat treatment, temperature, color change.

INTRODUCTION

Wood, as a renewable natural resource, has been used by humans for thousands of years. Since instability under changing moisture and biodegradability are major disadvantages of the material, considerable amount of research have been conducted on this topic. Heat treatment is a wood modification method being applied to improve dimensional stability and durability of the material (Esteves and Pereira, 2009). After heat treatment, the color of the wood changes, and the new color becomes uniform throughout the thickness of wood. Thus, thermal modification can be used to make a low-value wood to look like a high-value wood (Gunduz et al., 2009). In recent years, a broadening range of heat-treated softwood and temperate hardwood products have been marketed as alternatives to tropical hardwood for the external joinery and furniture applications. There are 30 companies across Europe operating thermal treatment plants with a total capacity of about 300,000 m^3. Approximately 40, 13, 12, 8% of this capacity is in Finland, Germany, Netherland and Estonia, respectively. The remaining is distributed in France, Croatia, Austria, Switzerland, Sweden and Turkey (Anonymous, 2011). Since there is no chemical application during the process, heat-treated wood is considered as an eco-friendly method which is an alternative to chemically impregnated wood materials (Rapp, 2001; Anonymous, 2003). A good literature review on wood modification by heat treatment, history and methods is done by Esteves and Pereira (2009). Depending on the variation of all heat treatment processes (industrial-scale, semi industrial scale or laboratory experiments), the properties of heat treated wood also vary. The extent of change in wood properties during heat treatment mainly depends on the heat treatment method, tree species and its wood characteristics, initial moisture content of the wood, the surrounding atmosphere and treatment temperature and time. Temperature has greater influence on many properties than time (Mitchell, 1988; Rapp, 2001; Hill 2006; Esteves and Pereira, 2009). ThermoWood, developed by VTT in Finland, is an industrial-scale heat treatment process that has been widespread in the market. With high temperatures of this treatment, bending strength is reduced to 30%. A considerable (from 50 to 90%) reduction in shrinking and swelling is reported after the treatment (Homan and Jorissen, 2004; Sahin Kol, 2010).

The main purpose of this study was to determine industrial heat treatment effects on some physical properties of *Pinus nigra* wood under varying temperatures and durations, which would give an opportunity for reduction of the process time.

MATERIALS AND METHODS

Wood samples used for this study were obtained from an industrial plant. Lumbers were stored and pre-dried at lumberyard in the conditions of 49% average relative humidity and 25°C air temperature. The thermal treatment was carried out in a prototype furnace of a forest product company following an industrial process called ThermoWood. Since the collaborated company has strict rules to keep the details of the process, only general mechanism of the process was mentioned here. In the process, wood was heated in the conditions of low air contents (under 3.5%) and in the presence of water steam. These conditions prevent wood material from burning at high temperature. Temperatures for the process range from 150 to 240°C, the time of duration range from 0.5 to 4 h (Homan and Jorissen, 2004). The temperature used for the actual heat treatment period is 212°C and duration of such temperature is 120 min for softwood species in the company where lumbers were planed, saw and test samples from sap wood were obtained. Control samples were cut from half of the lumbers. Heat treatment was applied at four different temperatures (190, 200, 212 and 225°C) and at three different duration (60, 120 and 180 min). Density of samples was measured according to ISO standard (ISO/CD 13061-2). As a result of the fact that wood density is a highly variable property between and within trees, the use of decrease density in percent to evaluate treatment effects was preferred. Percentage decrease in density was calculated by using control samples of each treatment. Dimensional stability was determined by measuring the volumetric swelling percentage of the samples which were immersed in a water bath at a controlled temperature of 20°C. During the tests, the water in the water bath was re-circulated continuously to maintain the required temperature of 20°C (Dubey, 2010). Swelling measurements of the samples were measured with 0.01 mm accuracy at three different marked positions before and after immersion in water for 2, 4, 8, 24, 48 and 72 h. Then, the specimens were weighed every 24 h and, once the weight changes of the test specimens were less than 0.1%, it was assumed that the specimens had reached equilibrium. The weight was measured with an accuracy of ±0.001 g. The dimensions in longitudinal, width (tangential) and thickness (radial) directions were measured to an accuracy of ±0.01 mm. The volumetric swelling coefficient (S) was determined by Equation (1) (Rowell and Youngs, 1981) given below:

$$\mathrm{S}\,(\%) = \frac{V2 - V1}{V1} x100 \qquad (1)$$

Where, V2 is the wood volume after wetting with water; V1 is the wood volume of oven dried sample before wetting.

Three replicates of untreated and treated specimens were used for each test. A variety of terms is used to describe the degree of dimensional stability given to wood by treatments: Antishrink efficiency, swelling percent, dimensional stabilization efficiency, antiswelling efficiency and percent reduction in swelling (R). We used antiswelling efficiency (ASE) and it was determined using the following Equation (2) (Rowell and Youngs, 1981):

$$\mathrm{ASE}\,(\%) = \frac{S2 - S1}{S1} x100 \qquad (2)$$

Where, S2 is the treated volumetric swelling coefficient; S1 is the untreated volumetric swelling coefficient.

Color of samples was measured according to ISO 7724/1-2-3 the tangential surface before and after heat treatment by a Minolta Croma-Meter CR-400 colorimeter. The sensor head was 6 mm in diameter. Measurements were made using a D65 illuminant and a 10-degree Standard observer. Percentage of reflectance, collected at 10-nm intervals over the visible spectrum (from 400 to 700 nm), was converted into the CIELAB color system, where L* describes the lightness and a* and b* describe the chromatic coordinates on the green-red and blue-yellow axes, respectively. From the L*, a* and b*values, the difference in the lightness (ΔL*) and chroma coordinates (Δa* and Δb*) were calculated using group mean values. ΔL*, Δa* and Δb* are the changes between pre and post treatment values. These values were used to calculate total color change (ΔE*) according to Equation (3) (ISO 7724/3):

$$\Delta E_{ab}* = [(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2]^{1/2} \qquad (3)$$

Variance analysis was applied for the results of this study. All statistical calculations were based on the 95% confidence level. Tukey's multiple range tests was used to determine the differences among treatments. Considering variations of four different temperatures, three process durations and particularly six different immersion times in water for the determination of swelling, the results were preferably given with simplified and understandable figures or tables and the important statistics were indicated in the text. Heat treated lumbers were evaluated visually in terms of quality in particular cracks.

RESULTS AND DISCUSSION

When compared with the control sample, wood density decreased with increasing time and temperature. The result is parallel to previously published report for different species (Ghalehno and Nazerian, 2011; Gunduz et al., 2009, Kaygin et al., 2009; Korkut and Guller, 2008; Yildiz et al., 2006). The highest decrease (12.6%) in density occurred at 225°C and 180 min, the lowest one (2.57%) was at 190°C and 60 min (Table 1).

According to statistical test on variation estimates for wood density decrease, temperature (62%) and duration (26%) are important sources of variations. On the other hand, the magnitude of temperature x duration interactions (5%) has lower importance than individual effects on the variation of wood density decrease. It means treatment temperature is more effective than the time duration and this result is consistent with some previous studies (Mitchell, 1988; Rapp, 2001; Hill 2006; Esteves and Pereira, 2009). The conditions of treatment differ among published works. Instead of extending this paper by comparing previous works which applied different conditions, a realistic point of view and focus on the studies including at least close conditions and/or species was done. For 190°C and 180 min duration, the density decrease of hornbeam wood was 4.88% by Ghalehno and Nazerian (2011) which is very close to that of the current result. However, at the same temperature and duration, and similar process for Scots pine, the result was reported to be about 3% (Kortelainen et al., 2006). The material of the reference works was 70°C kiln dried;

Table 1. Decrease (%) in *P. nigra* wood density after heat treatment.

Duration (min)	Temperature (℃)	N	Mean	Standard error
60	190	40	2.567	0.300
	200	40	2.638	0.105
	212	40	6.451	0.086
	225	40	8.194	0.172
120	190	40	4.457	0.576
	200	40	4.821	0.034
	212	40	8.150	0.175
	225	40	10.698	0.192
180	190	40	5.244	0.490
	200	40	7.837	0.369
	212	40	8.759	0.164
	225	40	12.602	0.303

on the other hand, samples of the current study was naturally pre-dried in different conditions and this may be the probable cause of the difference between the results. For paulownia wood at 200℃ and 180 min duration, density lose was found to be approximately 10% (Kaygin et al., 2009) which is 2% higher than that of our result. Akyildiz et al. (2009) reported approximately 13% density lost (12.7 for oven dry and 13.4 for air dry density) for *P. nigra* heat treated wood under 230℃ and 8 h. The result is similar to this study result (12.6%) of 225℃ and 180 min. The depolymerization reactions of wood polymers are the main cause of the density decreases. Above a certain temperature, the physical characteristics of hemicelluloses (127 to 235℃), lignin (167 to 217℃) and the cellulose (231 to 253℃) changes (Boonstra et al., 2007). Hemicellulose, which is less stable to heat effect than cellulose and lignin, plays important role in the decrease of physical properties of wood at high temperatures (Fengel and Wegener, 1989; Hillis, 1984).

As compared to the control group, heat treatments resulted in a significant ($P < 0.05$) reduction in swelling percent of wood, especially long time durations in water. The general trend is that volumetric swelling of wood decreases with increasing treatment temperature and durations (Figure 1). When compared with both the actual time and duration (212℃ and 120 min) in practice and suggested ones (230℃ and 120 min) which were obtained in laboratory conditions by Akyildiz et al. (2009), the current promising results of higher temperature (230℃) with a shorter duration (60 min) would present a possibility of having time reduction in practice for *P. nigra*.

Anti swelling efficiencies (ASE) of samples are shown in Figure 2. The highest ASE of 56 to 66% was found in the samples treated at 225℃ for 180 min. The second one was 225℃ for 120 min. The lowest ASE was found in 190℃ for 60 min (7 to 13%). The standard errors of means ranged from 0.13 to 0.71 among treatments. According to multiple comparison test results, there was no statistical difference ($P<0.005$) in the ASE for some treatments, that is, between 225℃, 60 min and 212℃, 120 min; 200℃, 60 min and 190℃, 120 min with no quality difference according to visual interpretation. This clearly indicates that treatment temperature is more effective on ASE than duration, especially treatment temperature of over 200℃. Dimensional stability is an important property of wooden material, particularly for the use under high humidity conditions. Thus, many studies have been conducted on this topic. Reported results on wood stability reductions (%) vary from one study to another one. These changes may explain differentiation of the heat treatment methods, standards and wood species. However, the general consensus is that heat treated wood has an advantage in terms of wood stability as compared to untreated ones (Hillis, 1984; Viitaniemi, 1997; Akyildiz and Ates, 2008; Korkut and Guller, 2008; Kaygin et al., 2009; Sahin Kol, 2010; Karlsson et al., 2011; Aydemir et al., 2011; Poncsac et al., 2011)

The availability and/or accessibility of the free hydroxyl groups of the wood play an important role in water sorption (Boonstra and Tjeerdsma, 2006). Degradation of carbohydrates and especially hemicelluloses causes a reduction in available free polar adsorption sites mainly of hydroxyl groups, including the free hydroxyl groups of water (Burmester, 1975; Feist and Sell, 1987; Hillis, 1984; Kartal et al., 2007; Aydemir et al., 2011). Irreversible hydrogen bond in the course of water movements within the pore system of the cell walls (Borrega and Karelampi, 2010) increase in the relative proportion of the crystalline cellulose, in which the hydroxyl groups are not easily accessible to water molecules (Pott, 2004) and cross linking of the lignin network (Tjeerdsma et al. 1998) might hinder the accessibility of free hydroxyl groups to

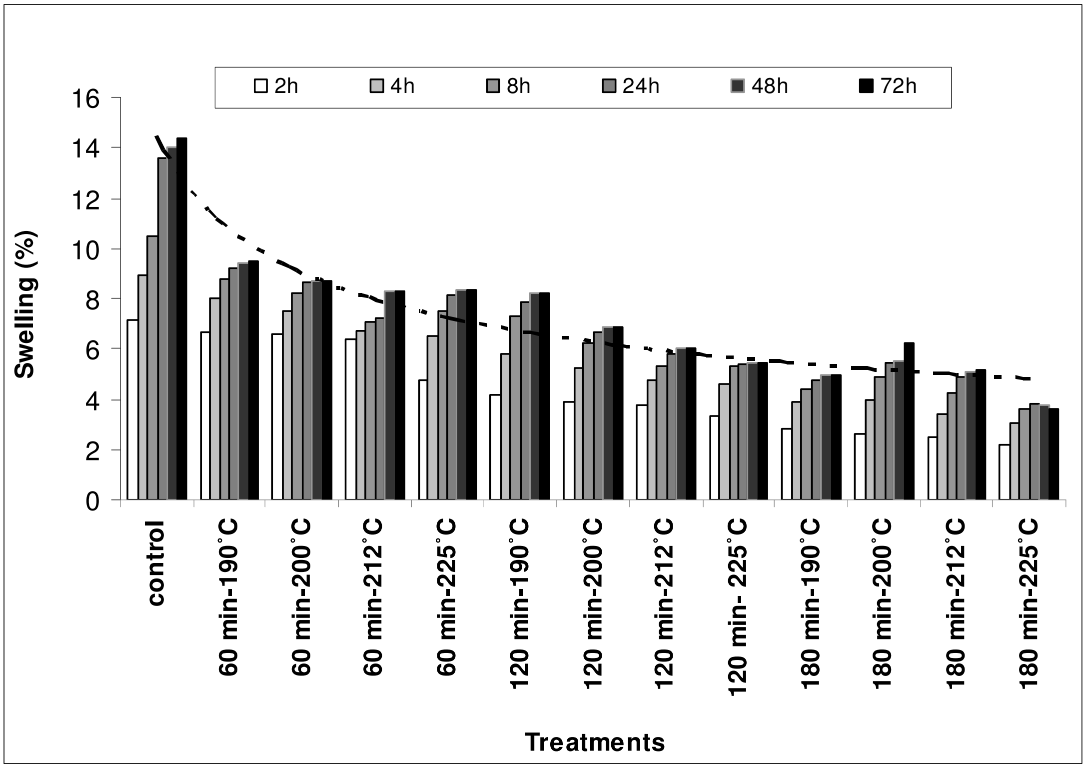

Figure 1. Swelling (%) of heat treated and untreated wood samples

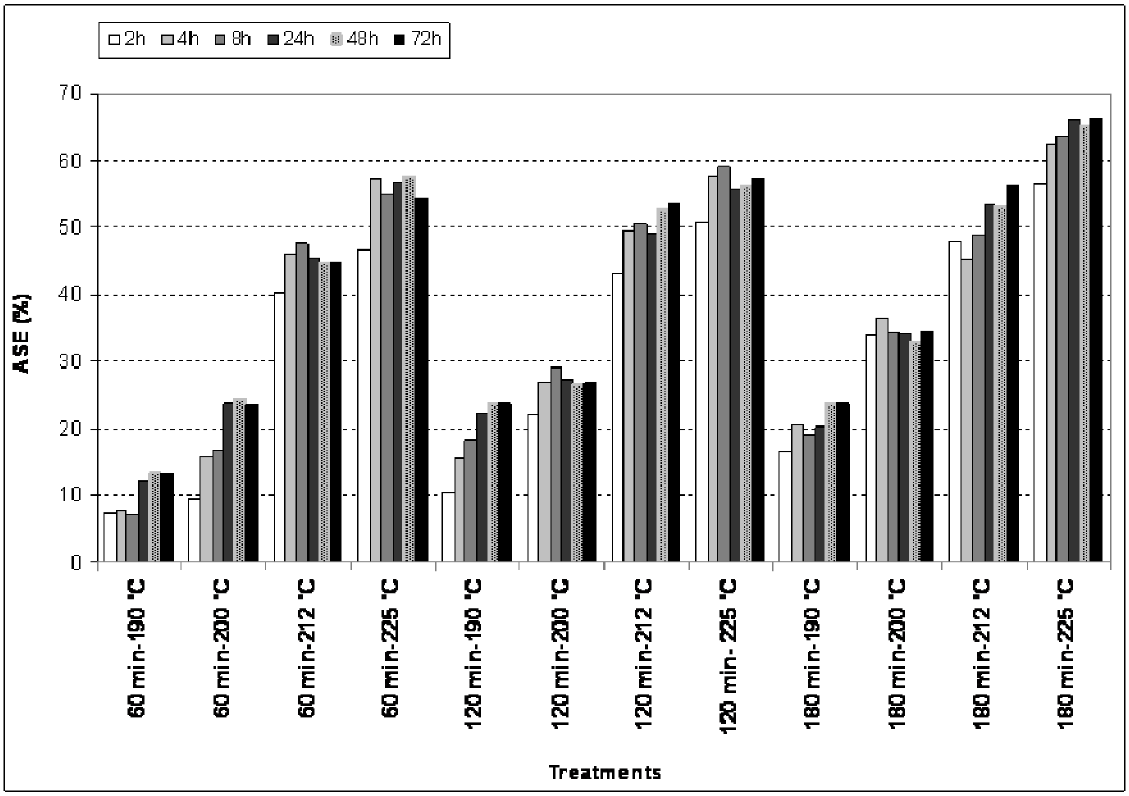

Figure 2. Antiswelling efficiency (ASE) of heat treated and untreated wood samples

Table 2. Effect of heat treatment on color properties of *P. nigra*.

Temperature (℃)	Duration (min)	L*	a*	b*	ΔL*	Δa*	Δb*	ΔE*
Control	-	67.849	12.960	18.911				
190	60	54.921	11.113	18.791	-12.928	-1.847	-0.120	13.060
	120	47.793	11.511	17.150	-20.056	-1.449	-1.641	20.175
	180	44.197	11.237	15.934	-23.652	-1.723	-2.857	23.886
200	60	42.719	10.322	15.141	-25.130	-2.638	-3.650	25.530
	120	37.866	10.947	13.362	-29.983	-2.013	-5.429	30.537
	180	34.286	9.833	10.976	-33.563	-3.127	-7.815	34.602
212	60	41.040	9.354	12.871	-26.809	-3.606	-5.920	27.691
	120	36.610	9.672	13.312	-31.239	-3.288	-5.479	31.886
	180	33.616	9.234	12.944	-34.233	-3.726	-5.847	34.928
225	60	31.750	9.824	11.558	-36.099	-3.136	-7.233	36.950
	120	29.610	8.738	10.010	-38.239	-4.222	-8.781	39.461
	180	23.165	4.255	5.195	-44.684	-8.705	-13.596	47.511

water (Pizzi et al., 1994). Furthermore, at very high temperatures (over 200℃), hemicelluloses may be changed to less hygroscopic substances like furfural polymers (Kamdem et al., 2002). Therefore, improved dimensional stability and water repellency of heat-treated wood are mainly due to decomposition or transformation of hemicelluloses at high temperatures.

The samples treated at 225℃ were found to be darker than the other samples. The color values for the different temperatures showed clear effect of temperature on color changes. In this study, L*, a* and b* values decreased after heat treatments. The highest and lowest decreases in L* were found for the treatments at 225℃ for 180 min and 190℃ for 60 min (Table 2). The negative value of lightness (ΔL*) and chromaticity coordinates (Δa*and Δb*) indicated that color became darker with increase in temperature and durations. According to Fengel and Wegener (1989) and Sundqvist (2002), the reason for color changes is the production of chromospheres as a result of the hydrolytic reactions that occur during heat treatment. The extent of thermal degradation is directly related to the extent of the darkening of the color properties (Kawamura et al., 1996). The extent of change in color to black for the treatment of 225℃ and 180 min, is an important aspect to consider in applications where the esthetic properties are of importance. Longer duration of time and/or higher temperature gives the wood a darker color. Therefore, the color can be used as an indicator of the severity of process conditions. However, the attained darker color after heat treatment is not stable against light exposure (Mitsui et al., 2003) and there has not been any cost-effective and easy method described to prevent this fading (Kaygin et al., 2009).

Conclusions

Depending on the time and temperature, heat treatment caused decrease (2.57 to 12.6%) in wood density of *P. nigra*; on the other hand it caused considerable gain (up to 66%) in wood stability, especially for long time immersions (more than 24 h) in wetting conditions. Results show that it is possible to have similar wood stability, applying high temperature and shorter time duration instead of lower temperature and longer durations for *P. nigra*. Longer heating time and high temperatures increase significantly the cost of the process. Any reduction in the duration of the thermal process without any deterioration of wood quality saves energy and increases productivity. In this study, only visual comparisons were made to compare samples quality after treatments. So, more detailed investigations may be required in the aspects of wood quality and energy.

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